

Report DRXTH-FS-TR-81106

SURVEY OF HAZARDOUS/CHEMICAL AREA NO. 2 OF THE  
FORMER ST. LOUIS ORDNANCE PLANT

VOLUME II

William R. Colvin  
Joseph H. Zarzycki

June 1981

FINAL REPORT

Distribution limited to US Government Agencies:  
Evaluation of Another Command: June 1981  
Requests for this document must be referred to:  
Commander, USATHAMA, ATTN: DRXTH-ES, APG-EA, MD 21010

US Army Toxic and Hazardous Materials Agency  
Aberdeen Proving Ground, MD 21010

40330678



Superfund

#### DISCLAIMER

The views, opinions and/or findings contained in this report are those of the authors and should not be constructed as an official Department of the Army position, policy, or decision, unless so designated by other documents.

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Report DRXTH-FS-TR-81106	2. GOVT ACCESSION NO. ---	3. RECIPIENT'S CATALOG NUMBER ---
4. TITLE (and Subtitle) Survey of Hazardous/Chemical Area No. 2 of the Former St. Louis Ordnance Plant  Volume II		5. TYPE OF REPORT & PERIOD COVERED Final Report
		6. PERFORMING ORG. REPORT NUMBER ---
7. AUTHOR(s) William R. Colvin Joseph H. Zarzycki		8. CONTRACT OR GRANT NUMBER(s) ---
9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Toxic and Hazardous Materials Agency Aberdeen Proving Ground, MD 21010		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS ---
11. CONTROLLING OFFICE NAME AND ADDRESS US Army Toxic and Hazardous Materials Agency Aberdeen Proving Ground, MD 21010		12. REPORT DATE June 1981
		13. NUMBER OF PAGES 272
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Distribution limited to US Government Agencies: Evaluation of Another Command: June 1981 Requests for this document must be referred to: Commander, USATHAMA, ATTN: DRXTH-ES, APG-EA, MD 21010		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) ---		
18. SUPPLEMENTARY NOTES Volume II contains only the appendices for the Survey Report (Volume I)		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Volume II St. Louis Ordnance Plant Goodfellow US Army Reserve Center Hanley Industries US Army Toxic and Hazardous Materials Agency		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This volume contains the following appendices to Volume I of the survey report: Appendix A: Authority Appendix B: Archives Search Appendix C: Description of Buildings in the GUSARC and Hanley areas. Appendix D: Tables of Complete Analytical Results  (Continued on next page)		

Continued from Block 20:

Appendix E: Analytical methods and Quality Assurance Data for the Goodfellow US Army Reserve Center Survey

Appendix F: Analytical Methods and Quality Assurance Data for the Hanley Area Survey

Appendix G: Sample Standard Operating Procedure. "Demolition Plan for the 400 Area (Explosives Manufacturing Area) of the Frankford Arsenal"

**Survey of Hazardous/Chemical Area No. 2  
of the Former St. Louis Ordnance Plant**

**VOLUME II**

## LIST OF APPENDICES

- APPENDIX A: Authority
- APPENDIX B: Archives Search
- APPENDIX C: Description of Buildings in the Goodfellow US Army Reserve Center and Hanley Areas
- APPENDIX D: Tables of Complete Analytical Results
- APPENDIX E: Analytical Methods and Quality Assurance Data from the Goodfellow US Army Reserve Center Survey
- APPENDIX F: Analytical Methods and Quality Assurance Data for the Hanley Area Survey
- APPENDIX G: Sample Standard Operating Procedure "Demolition Plan for the 400 Area (Explosives Manufacturing Area) of Frankford Arsenal"

**APPENDIX A**

**AUTHORITY**



**DEPARTMENT OF THE ARMY**  
**OFFICE OF THE CHIEF OF ENGINEERS**  
**WASHINGTON, D.C. 20314**

REPLY TO  
ATTENTION OF:

**DAEN-RE:1-C**

**18 December 1978**

**SUBJECT: Installation Restoration, Goodfellow U. S. Army Reserve Center  
(Portion of Former St. Louis Ordnance Plant), Missouri**

**Commander  
U. S. Army Materiel Development  
and Readiness Command  
ATTN: DRCDNR  
5001 Eisenhower Avenue  
Alexandria, Virginia 22333**

1. A 27.88 acre parcel of property which was part of the former St. Louis Ordnance Plant has been determined excess to the needs of the Army. The Department of Labor (DOL) is interested in acquiring the property for the establishment of a Job Corps Training Center. DOL has requested authorization to immediately initiate modification of certain of the buildings.
2. Approximately 13.20 acres of the area was last used as a Reserve Training Center with a 14.68 acre portion being leased to Hanley Industries, Inc., for the manufacturing of detonators and other explosive devices. Since DOL will use the property on an unrestricted use basis, certification that it is free from hazardous materials and/or contamination is required. Certification falls within the purview of the Installation Restoration Program.
3. Request expeditious actions be taken to determine the status of the property with respect to contamination and that a schedule for the determination be provided this office.
4. Because of DOL need for earliest possible use of the 13.20 acre area, a separate certification should be provided thereon, and, certification relative to the remaining 14.68 acre area should be provided at an early date thereafter.

✓  
11771

DAEN-REM-C

18 December 1978

SUBJECT: Installation Restoration, Goodfellow U. S. Army Reserve Center  
(Portion of Former St. Louis Ordnance Plant), Missouri

5. Coordination of the transfer of property with DOL will be the responsibility of Mr. W. Jaster of this office, Autovon Telephone No. 223-6175.

FOR THE CHIEF OF ENGINEERS:



JAMES E. UELMEN  
Acting Chief, Military Branch  
Management & Disposal Division  
Directorate of Real Estate

Ss 10 Jan 78

DRCIS-EP (18 Dec 78) 1st Ind

SUBJECT: Installation Restoration, Goodfellow U. S. Army Reserve Center  
(Portion of Former St. Louis Ordnance Plant), Missouri

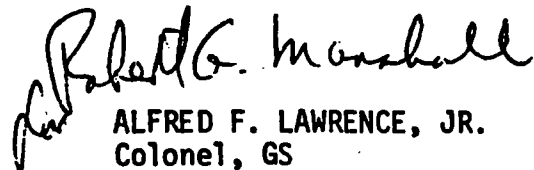
HQ, US Army Materiel Development & Readiness Command, Alex, VA 22333 26 Dec 1978

TO: Project Manager for Chemical Demilitarization and Installation Restoration,  
ATTN: DRCPM-DRR, Aberdeen Proving Ground, MD 21010

1. Reference telephone conversation between Mr. Andy Anderson, OPMCDIR, and  
Mr. Robert Marshall, DRCIS-EP, 26 December 1978.

2. Forwarded for appropriate action and information for a reply in accordance  
with paragraph 3 of basic correspondence. Request response not later than  
10 January 1979.

FOR THE COMMANDER:



ALFRED F. LAWRENCE, JR.  
Colonel, GS  
Chief, Engineer Division  
Directorate for Installations  
and Services

**APPENDIX B**  
**ARCHIVES SEARCH**

**ST. LOUIS ORDNANCE PLANT ARCHIVES SEARCH**

## TABLE OF CONTENTS

	<u>PAGE</u>
I. General	1
A. Purpose of the Archives Search	1
B. Authority	1
C. Introduction	1
II. Personnel/Agencies Contacted	1
III. Findings	2
A. Installation History 1941-1959	2
B. Activities in Hazardous Area No. 2, 1941-1945	5
C. Activities in Hazardous Area No. 2, 1945-1959	6
IV. Document Sources	6
A. Files Available at National Personnel Record Center, St. Louis, MO	6
B. Files Destroyed & Dates Destroyed, National Personnel Record Center, St. Louis, MO	6
C. Files Available at Washington National Records Center, Suitland, MD	7
V. Conclusions	7

LIST OF FIGURES

	<u>PAGE</u>
Figure 1 - Site Map of Hanley Area, St. Louis Ordnance Plant, MO	3

**ST. LOUIS ORDNANCE PLANT  
ARCHIVES SEARCH**

**I. General**

A. Purpose of the Archives Search: This historical summary was conducted to determine operations at the St. Louis Ordnance Plant (SLOP) during the years 1941-1959. Foremost in the search was historical information relative to the use and subsequent decontamination of the buildings and magazines associated with Hazardous Area No. 2/Chemical Area No. 2.

B. Authority: DF, Chief, Field Systems Division to Chief, Environmental and Safety Division, 25 June 80, subject: Request for Archives Search of the St. Louis Ordnance Plant - Hazardous Area No. 2/Chemical Area No. 2.

C. Introduction: The Hanley Area, a contractor operated portion of the Hazardous Area No. 2, is to soon be acquired by the Department of Labor. It is imperative that the history of this portion, prior to 1959, be known in order to identify those buildings and magazines where explosives and heavy metals were used. A history of the operations conducted in the Hanley Area exists for the period 1959 to present.

**II. Personnel/Agencies Contacted**

Personnel from the following agencies were contacted to provide information concerning the search:

- A. National Personnel Records Center, St. Louis, MO
- B. Industrial and Social Division of the National Archives, Washington, D.C.
- C. Washington National Records Center, Suitland, MD
- D. Environmental Photographic Interpretation Center, Vint Hill Farms Station, Warrenton, VA
- E. U.S. Army Corps of Engineers, Kansas City District, MO
- F. Retired SLOP Post Engineer, St. Louis, MO
- G. Department of Agriculture, ASCS Imagery, Bionetics Corporation, Vint Hill Farms Station, Warrenton, VA
- H. Commander's Representative for St. Louis Army Ammunition Plant (SLAAP), St. Louis, MO
- I. Retired Commander's Representative for SLAAP, St. Louis, MO
- J. Plant Manager for SLAAP, St. Louis, MO

K. Data Processing and Information Management Branch of Administrative Services Division, US Army Environmental Hygiene Agency (USAEHA), APG, MD

L. US Army Armament Materiel Readiness Command (ARRCOM) Historical Office, Rock Island, IL

### III. Findings

#### A. Installation History 1941-1959

The following summary was taken from internal documents of the St. Louis Ordnance Plant files and from Historical Report, 45-55, St. Louis Ordnance Plant, primarily using Chevrolet Motor Division records when plant files were unavailable.

Construction was started during January 1941 and was completed in May 1942. Initial production, however, started as early as December 1941. During the World War II period, the St. Louis Ordnance Plant was constructed and activated as a Government Owned-Contractor Operated (GOCO) plant for the production of small arms ammunition (caliber .30 and .50) and components for the 105 mm shells.

Major contractor operators during the subject period were:

1. The United States Cartridge Company (U.S. Cartridge), a subsidiary of Olin Industries, operated the ammunition plant for manufacture of small arms ammunition and produced 67 million rounds. U.S. Cartridge occupied plants No. 1 and No. 2. Plant No. 1 comprised buildings in the 100 series and Plant No. 2 the 200 series (see Figure 1, page 3). They are both located due east of Bldg 107.

2. The McQuay-Norris Manufacturing Company operated the core plant for manufacture of cores for small arms ammunition and produced 8,500 million cores. McQuay-Norris occupied the 113 building series on the extreme south end, located south of Highway 70 on Goodfellow Boulevard near Natural Bridge Road.

3. The Chevrolet-Shell Division of the General Motors Corporation operated the shell plant for the manufacture of 105 mm shells. This facility began production late in 1945 following the changeover from small arms ammunition manufacture. The number of shells produced is unknown.

Following deactivation of the plant in 1945, all property and buildings were transferred to the Seventh Service Command for use as an administrative center for the Army Service Forces. This included the plants No. 1 and No. 2 areas, but excluded the McQuay-Norris plant.

During the period 1945 to 1951 the Plant was utilized as a record center for maintaining and servicing records of the Adjutant General's Office and the Finance Center, US Army. During this period the Plant was administered by the St. Louis Administration Center, a Class I activity under the jurisdiction of Fifth Army Region.

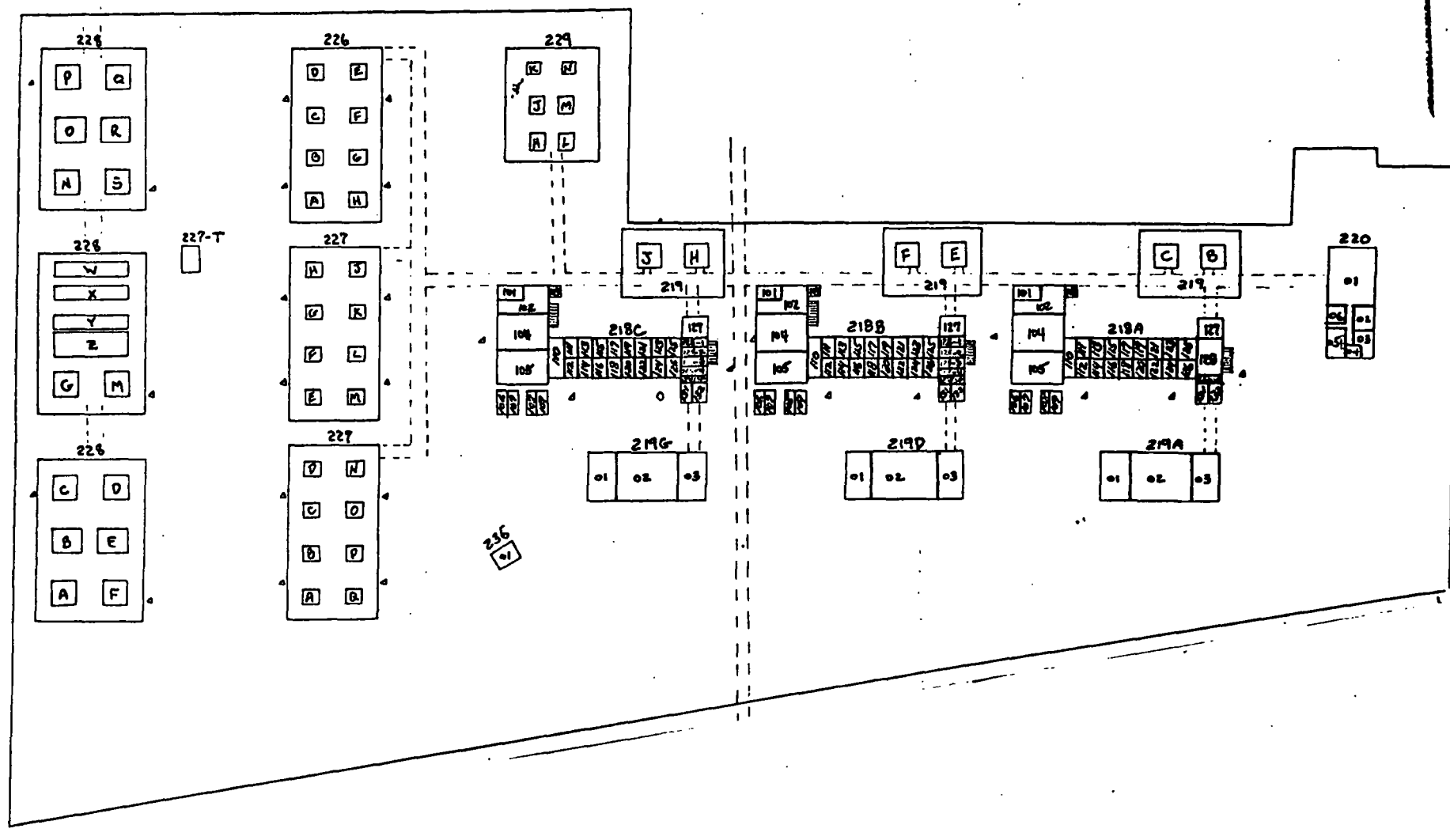


FIGURE 1. SITE PLAN OF THE HANLEY AREA OF THE FORMER ST.

LOUIS ORDNANCE PLANT, ST. LOUIS, MISSOURI

Between 1945-51, seven Government housing buildings were constructed, each containing four apartments. In addition, a Wherry Housing Project consisting of 16 buildings containing a total of 120 apartments was completed.

On 1 Aug 1951, the Plant was established as a Class II industrial installation under the jurisdiction of the Chief of Ordnance and placed in an active status under command of the Ordnance Small Arms Ammunition Center. On 1 Sep 1951, the St. Louis Core Plant was redesignated from a Class II industrial installation to a Class II industrial activity at this Plant.

On 1 Feb 1952, the Chevrolet-Shell Plant was discontinued as a Class II industrial installation and its facilities consolidated with and made a part of the St. Louis Ordnance Plant. On the same date the St. Louis Core Plant was discontinued and its facilities were also consolidated with and made a part of the St. Louis Ordnance Plant.

Three divisions of SLOP were established:

1. Ammunition Division - Small arms ammunition manufacturing activities; contractor-operator U.S. Defense Corporation.
2. Core Division - Formerly St. Louis Core Plant; contractor-operator McQuay-Norris Manufacturing Company.
3. Shell Division - Formerly Chevrolet Shell Plant; contractor-operator Chevrolet Shell Division.

On 1 May 1952 the St. Louis Administration Center was discontinued and its function transferred to the St. Louis Ordnance Plant. These functions consisted of Army housekeeping services to Army Finance Center. In view of the Plant mission, efforts were immediately started to eliminate the housekeeping functions and by the end of September 1953, they were eliminated to the extent that the Army Service Unit was discontinued and the services decentralized to other Fifth Army Agencies within the St. Louis area.

As of 1 July 1954, this plant was placed under the command supervision of the US Army Ordnance Ammunition Command vice the US Army Ordnance Small Arms Ammunition Center which was being disbanded.

Facilities formerly operated by Chevrolet Shell Division of General Motors Corporation were placed in standby under power status, 30 Aug 1954. From 1951 through 1954 a cumulative total of 19,094,321 shells were produced. Unlike the shells of WW II era production, these shells were phosphate coated.

Later activities consisted of manufacturing small arms ammunition by the United States Defense Corporation and small arms ammunition cores by the McQuay-Norris Manufacturing Company.

Effective 1 Dec 1957, the St. Louis Ordnance Plant, a Class II industrial installation of the Chief of Ordnance, was placed in an inactive status.

## B. Activities in Hazardous Area No. 2, 1941-1945

According to information provided by a former employee, of the 37.35 acres comprising Hazardous Area No. 2, the portion on the north end, specifically Bldgs 223 A and B, used magnesium for tracer bullet manufacture. In Bldg 234, on the extreme south end, lead styphnate and possibly tetrazine were used in the manufacture of primers.

The former employee recalled that areas designated on site map of Hanley Area, page 3, were used prior to 1945 as follows:

<u>AREA</u>	<u>USE</u>
220	Offices
218 A,B,C	Primer & Tracer Mixing
219 A,D,G	Primer & Tracer Mixing
226, 227	Remote Mixing Operation
228	Powder Storage

The following information was taken from documents entitled Disposition of the St. Louis Ordnance Plant and from Chapter I - Major Activities, Mopping Up, a formerly restricted publication of unknown origin found in the plant files.

In 1945, following deactivation procedures, machines having fire and explosive hazards due to collection of powders in hidden parts and/or places on the machines and buildings having explosive hazards were decontaminated by the Army COE in accordance with regulations of the Safety and Security Branch, Office, Chief of Ordnance, Chicago. The decontamination included machines being scrapped as well as those being disposed of through other channels. All special equipment peculiar to the production of small arms ammunition was declared excess and recommended for scrapping. All multipurpose equipment was reported to the Reconstruction Finance Corporation for ultimate disposition. Over 14 million pounds of scrap material were moved from the plant.

The War Department Supply Bulletin, SB 5-52, July 1945, Decontamination Procedures, outlined the procedures for decontamination or discontinuation of operations prior to dismantling, demolition, alteration, or conversion of plants or equipment or areas where explosives or toxic materials have been manufactured, and in other plants and areas where similar hazards exist. A system of markings used to show extent of decontamination ranges from a simple "X" to indicate material only partially decontaminated to five "X" indicating safe. A review of SB 5-52 by an Explosive Ordnance Disposal Officer assigned to Environmental and Safety Division of the US Army Toxic and Hazardous Materials Agency revealed that procedures had changed little from the date of publication to present. Procedures were stringent, including detailed cleaning with water, steam, or

solvents, neutralization of acids, and flashing or burning of contaminated equipment to reach a "XXXXX" level of decontamination. Current procedures are listed in ARROOM 385-5, dated 26 Oct 1979.

The buildings and magazines in the entire Hanley Area were marked "XXXXX" which indicated that the structures had been inspected after decontamination and were considered safe, and that no explosives remained in the structures.

One former government employee, a plant maintenance engineer who often served as Post Engineer, confirmed the specific documenting of decontaminations. However, copies of the COE completion report have not been located and it is assumed that none survived as archival documents. Accordingly, no record of toxic or hazardous activities from 1945 until the Hanley lease of 1959 were uncovered.

#### C. Activities in Hazardous Area No. 2, 1945-1959

As buildings were available, the Finance Center conducted classes in this area until 1951. In 1951, a rehabilitation program for small arms ammunition manufacture was begun in the area. The machinery was installed but production was never begun. After the Korean Conflict, the machinery was removed and disposed of. It was reported that at various times the area was used for storage and for automotive and equipment maintenance.

#### IV. Document Sources

##### A. Files Available at National Personnel Record Center, St. Louis, MO

###### File Container No.

- 05-25-52-7-3 Memos, General Orders, Special Orders, Bulletins, Procedural Standardization Files
- 06-25-04-2-1 Minutes of Staff Meetings, Minutes of Ind. Planning Board, Memos, Bulletins, Special Orders, SOP's
- 08-41-10-E-3 Trip Reports, Newspapers, Clippings, Photos

##### B. Files Destroyed and Dates Destroyed, National Personnel Record Center, St. Louis, MO

1951-1957 Files of Government-Owned, Contractor Operated Industrial... Destroyed 1 July 1964

Unserviceable Scrap... Destroyed 1976

Contract Files Over \$25,000. Destroyed 1963

1951-1952 Plant General Administrative Files - Could not be located.

**MD C. Files Available at Washington National Records Center, Suitland,**

**Record Group 156, Entry 646, Box 210 - Decontamination and  
Equipment Disposition, Newspaper Clippings**

**Record Group 156, Entry 646, Boxes A560 and 558 - St. Louis  
Ordnance District History, Volumes I - XIV**

**V. Conclusions**

**A. Documents specific to land transfers, buildings and magazines requiring heavy metal use, explosive use, or decontamination reports were not located during the subject search.**

**B. Information presented from personal interviews may prove useful relative to general locations and recalled plant operations. This information was not corroborated by other sources.**

**APPENDIX C**

**Description of Buildings in the  
Goodfellow US Army Reserve Center and Hanley Areas**

1. Descriptions of Buildings and Bunkers in the Goodfellow US Army Reserve Center Area

a. Building 234

This large "U" shaped building has an east and west wing comprised of red painted cement floors with glazed brick walls. The central part of 234 has a central (northern room) adjoined by (going south) a room with metal cages, a large room with some old shower stalls, a kitchen, mess hall, and some smaller rooms including a toiletroom.

The southwestern part of the building has a floor constructed of wooden blocks. A mezzanine (very small) exists in the western part of the building. There is also a basement which houses the heating system in the north central part of this building.

b. Building 223A

Building 223A is a large rectangular, one-story building whose length runs north-south and its width runs east-west. One large room on the first floor is divided up into 21 sections on the east and 21 sections on the west sides of the building. These sections are referred to as bays in the southern portion of the building. The surface of the first floor is red painted cement. The walls are all glazed brick. Each section has six overhanging fluorescent lights. The basement of the building has five (5) large heating units.

c. Building 223B

Building 223B is characteristically the same as 223A except that it is smaller, having only 13 bays on either side. It also runs north-south lengthwise, parallel to and directly across the street from 223A. The walls are glazed brick. The northern half of the building floor is painted red. The southern half is basically unpainted except for the bays between walls 10C and 11C and 10B and 11B. The floor in the southern half may have been painted at one time--some signs of red paint are indicated. The ceiling is about 25 ft. high, similar to 223A. A basement exists where five heating units similar to those in 223A are housed. This is beneath the northwestern section of the building. Three pollution control systems enclosed in three houses are on top of 223A and 223B.

d. Building 222A

This building is a one-story, irregularly-shaped, rectangular structure with an unpainted floor. Building 222A has many rooms including toiletrooms and shower rooms. Walls are painted white (upper section) and green and blue (lower section). The basement is in the southern section of the building and contains the heating system. Three exhaust systems enclosed in houses are on top of the roof.

e. Building 231 Series

This series consists of 8 small one-room red brick buildings with glazed brick interiors and red painted floors.

f. Building 243 Series

The 243 series consists of 8 small, two-roomed, white buildings constructed of plasterboard and shingles. The floors are red-painted cement. Each building is surrounded by 15 foot high cement blast walls roughly 18 inches thick. The front part of the walk going into 243D is soaked with oil. Some oil barrels are stored in the area.

g. Building 232 Series

The buildings in 232 series are similar to those in the 243 series. Walls are constructed of plasterboard (interior) and green shingle (exterior). The floors are red painted cement. A partition (wall between rooms) is present in several of the buildings. Each building is surrounded by 15 foot high blast walls as is the 243 series. There are 8 buildings in this series.

h. Building 244

This building formerly housed a power plant. It has a basement and first floor consisting of one large room. First floor is contaminated with bird excrement.

i. Underground Bunkers

There are six underground bunkers which are located south of building 234. Each bunker is approximately 4 feet by 6 feet by 8 feet. There are cement steps leading to each bunker. The stairways are enclosed with a concrete canopy.

j. Bunker 224

This underground bunker is located Northwest of building 223B. Bunker 224 was accessed by a stairwell. Because the bunker was filled with water, it was not possible to determine its dimensions. However, the walled area above the bunker measured approximately 30 feet by 30 feet and the bunker appeared to be about 20 feet deep.

2. Descriptions of Buildings in the Hanley Industries Inc. Leased Area

a. Building 218 Series

Series 218 buildings are large T shaped buildings with a red brick exterior and glazed brick interior walls. Floors throughout the buildings are of anti-static composition construction. The south end consists of three large and six smaller rectangular rooms. The center portion contains 17 rooms, a large one on the south end and the smaller ones extending to the north end. The north end of buildings 218B and C contain eight rooms. Four are of poured concrete construction. The north end of building 218A consists of two large and two small rooms. All rooms open to the building exterior. Each room has overhanging fluorescent lights.

These buildings are interconnected by underground tunnels. Basement rooms are located at each end of the buildings. At the south end are four rooms which open into a foyer. One of these is an equipment room, the others appear to be storage rooms. At the north end is one large basement room connected to the tunnel system.

b. Building 219 Series

The buildings in the 219 series are of two types. Those to the east are large, single story, rectangular, brick buildings, whose length runs north-south. These consist of a large control room with smaller rooms at each end. All rooms have orange colored glazed brick interior walls, anti-static composition floors, overhanging fluorescent lights, and wall and ceiling vents. These buildings also have outside loading docks. There are no basements.

The 219-series buildings to the west (B, C, E, F, H, J) are square, one room brick buildings with orange colored glazed tile interior walls and anti-static floors. The ceilings are plasterboard and contain a vent. The roofs are clay tile. All of these buildings are surrounded by 15 foot high concrete blast walls.

c. Building 220

Building 220 is a large rectangular, one-story brick structure whose length runs east-west. This was a laboratory facility. The west end consists of one large room with laboratory benches at the east end and a laboratory hood in the center. The east end consists of a hallway and five rooms, two of which are restrooms. A stairwell at this end leads to the basement. All rooms on the ground floor have orange colored glazed brick interior walls, anti-static composition floors and overhead fluorescent lights.

The basement of building 220 is a single large room which was used as a machine shop. The walls and floors in the basement are concrete.

d. Building 226 and 227 Series

The 226 and 227 series consists of small one room, white buildings constructed of plasterboard interior walls, asbestos shingle outer walls, and anti-static compound floors. Each building is surrounded by 15 foot high concrete walls approximately 18 inches thick.

e. Building 227T

This is a temporary one story, one room building with plasterboard interior walls, asbestos shingle outer walls, with a solid concrete floor.

f. Building 228 Series

The buildings in this series are of two types. Those in the east and west groupings are square brick buildings with orange colored glazed brick interior walls and anti-static compound floors. The south central building in each grouping has a stairwell which leads to the underground tunnel network connecting all of the buildings.

The buildings in the center grouping (228W, 228X, 228Y) are long, rectangular structures constructed of plasterboard interior walls and asbestos shingle outer walls. The buildings house drying ovens. Two of the buildings (228G, 228M) in this grouping are square with brick exterior and orange colored glazed brick interior walls.

All of the buildings in this series have anti-static compound floors and are surrounded by 15 foot high blast walls as in the 226 and 227 series.

g. Building 229 Series

Buildings 229H, J, L and M are one room rectangular, underground, poured concrete structures with heavy steel vault doors. Each building has a vent in the center of the ceiling leading to the surface. Access to these buildings is by way of concrete stairways. The stairways and area above each structure are surrounded by thick, 15 foot high poured concrete explosion walls.

Buildings 229K and N are above ground, rectangular, of wood frame construction with exterior asbestos shingles. The interior walls are plasterboard. Each building contains a stainless steel sink with wall to wall drainboards. These buildings are also located within 15 foot high poured concrete explosion walls.

h. Building 236

Building 236 is a one story double garage of brick construction with a concrete floor.

**APPENDIX D**

**TABLES OF COMPLETE ANALYTICAL RESULTS**

**Table 1. Goodfellow US Army Reserve Center  
Results of Heavy Metal Analyses of Surface Samples**

Building	Sample #	Concentration, $\mu\text{g}/\text{m}^2$			
		Cd	Cr	Pb	Hg
222A	81	58.8	321.6	5775.	< .23
	82	29.8	150.8	3465.	3.02
	83	39.2	110.6	1995.	1.62
	84	29.4	170.8	4515.	2.24
	86	29.8	251.2	4200.	15.9
	87	29.4	130.6	3150.	1.48
	85 (Blank)	< .99	< 2.2	2.1	.23
223A	465	178.2	2575.	13650.	< .23
	500	120.	2678.	3360.	.85
	21	58.8	301.5	966.	.82
	22	5.97	50.2	1365.	.39
	23	5.	50.2	504.	.25
	24	19.6	261.3	4410.	.36
	25	844.2	462.3	3780.	.34
	26	98.	261.3	2625.	.51
	27	88.2	341.7	2594.	.46
	28	49.	1751.	5040.	.37
	29	98.	1648.	5775.	1.70
	40	49.	1009.4	36440.	2.13
	41	117.6	1648.	7875.	< .23
	42	148.5	2060.	13230.	.78
	43	117.6	2091.	5282.	.34
	45	49.8	291.4	2625.	.34
	46	58.8	160.8	2310.	3.47
	47	68.6	597.4	12600.	.34
	48	99.	5562.	2100.	.72
	49	68.6	381.9	2205.	.30
	60	58.8	236.9	2625.	1.59
	44 (blank)	< .99	< 2.2	21.2	< .23
223B	61	39.2	648.9	1890.	.41
	62	5.	20.1	724.5	< .23
	63	68.6	482.4	1470.	.23
	64	19.6	211.0	1029.	.52
	65	29.4	241.2	1470.	.44
	66	9.9	60.3	1575.	.30
	67	78.4	341.7	2205.	< .23
	69	4.98	10.0	474.7	< .23
	79	107.8	462.3	1260.	.44
	80	49.	180.9	2058.	1.17
	68 (blank)	< .99	< 2.2	1.1	< .23

Table 1 (Continued)

Building	Sample #	Cd	Cr	Pb	Hg
234	1	3.5	432.6	598.5	.52
	2	49.8	301.5	945.	11.66
	3	39.8	170.8	865.2	4.14
	4	29.8	351.8	2029.	7.62
	5	79.6	221.1	1754.	1.17
	6	783.9	2987.	2940.	13.44
	7	29.4	80.4	861.	1.34
	8	306.9	587.1	1365.	7.62
	9	168.3	463.5	3570.	6.16
	10	138.6	100.5	2205.	44.8
	12	79.6	1236.	2835.	3.81
	13	217.8	824.	1260.	7.73
	14	844.2	1648.	4515.	5.38
	15	4422.	4120.	14700.	4.7
	16	29.4	367.5	2100.	.34
	17	29.4	231.	1470.	.41
	18	6.0	60.3	1533.	<.23
	19	9.95	66.	903.	<.23
	20	58.8	515.	1680.	.28
	436	58.8	572.8	3360.	1.48
	11(Blank)	9.9	<2.2	52.	.41
244	147	148.5	710.7	6615.	<.23
	148	4.0	150.8	535.3	<.23
	149	29.4	422.1	2730.	.23
	88	148.5	422.1	3360.	<.23
	89	5.0	20.1	3465.	<.23
231A					
231C					
231E	501 (a)	49.	535.6	3150.	.23
231G					
231N					
231P					
243A	502 (a)	98.	762.2	5985.	<.23
243B					
243C					
243D	503 (a)	49.	1133.	15750.	<.23
243E					

Table 1 (Continued)

Building	Sample #	Cd	Cr	Pb	Hg
243F	504 (a)	58.8	906.4	7350.	<.23
243G					
243H					
232A					
232A	505 (a)	68.6	504.7	18900.	.26
232B					
232C					
232D					
232E	506 (a)	178.2	525.3	6720.	.23
232F					
232G					
232H					

(a) Composite sample.

## Thin Layer Chromatography Results

The results of the TLC analyses of physical and swab samples which gave positive responses are given in Table 2. The levels of TNR, RDX, TNT, NC, and Pb styphnate were all below their TLC detection limits.

In a few cases, PETN and NG were detected, but masked by an interfering material. Subsequent semi-quantitative analysis of these samples by HPLC confirmed the presence of these materials at or about the TLC detection limit. These samples were not rigorously quantitated by HPLC as this effort was beyond the scope of work for this program.

A positive response was obtained in the TLC analysis for tetrazene in several samples. However, these same samples also contained large amounts of interfering materials which contained reducible nitrate groups and masked the TLC visualization of NG and PETN. Bird manure (guano) is composed of a variety of nitrates and guanidine type structures, and it is probable that such compounds would give false-positive responses for PETN, NG and tetrazene. It is, therefore, believed that the positive responses for tetrazene were due to the presence of these interfering compounds and not due to the presence of tetrazene.

**Table 2. Goodfellow US Army Reserve Center  
Results of the Analyses of Physical and Swab Samples**

Sample No.	Building	TNR	RDX	TNT	NG	PETN	NC	Pb Sty	Tetrazene
160	231P	ND	ND	ND	ND	ND	ND	ND	ND
161	231P	ND	ND	ND	ND	ND	ND	ND	ND
162	231P	ND	ND	ND	ND	ND	ND	ND	ND
163	231N	ND	ND	ND	ND	ND	ND	ND	ND
164	231N	ND	ND	ND	ND	ND	ND	ND	ND
165	231N	ND	ND	ND	ND	ND	ND	ND	ND
169	231J	ND	ND	ND	ND	ND	ND	ND	ND
170	231J	ND	ND	ND	ND	ND	ND	ND	ND
171	231J	ND	ND	ND	ND	ND	ND	ND	ND
172	231G	ND	ND	ND	ND	ND	ND	ND	ND
173	231G	ND	ND	ND	ND	ND	ND	ND	ND
174	231G	ND	ND	ND	ND	ND	ND	ND	ND
175	231E	ND	ND	ND	ND	ND	ND	ND	ND
176	231E	ND	ND	ND	ND	ND	ND	ND	ND
177	231E	ND	ND	ND	ND	ND	ND	ND	ND
178	231C	ND	ND	ND	ND	ND	ND	ND	ND
179	231C	ND	ND	ND	ND	ND	ND	ND	ND
180	231C	ND	ND	ND	ND	ND	ND	ND	ND
181	231A	ND	ND	ND	ND	ND	ND	ND	ND
182	231A	ND	ND	ND	ND	ND	ND	ND	ND
183	231A	ND	ND	ND	ND	ND	ND	ND	ND
184	243H	ND	ND	ND	ND	ND	ND	ND	ND
185	243H	ND	ND	ND	ND	ND	ND	ND	ND
186	243H	ND	ND	ND	ND	ND	ND	ND	ND
187	243G	ND	ND	ND	ND	ND	ND	ND	ND
188	243G	ND	ND	ND	ND	ND	ND	ND	ND
189	243G	ND	ND	ND	ND	ND	ND	ND	ND
190	243F	ND	ND	ND	ND	ND	ND	ND	ND
191	243F	ND	ND	ND	ND	ND	ND	ND	ND
192	243F	ND	ND	ND	ND	ND	ND	ND	ND
193	243E	ND	ND	ND	ND	ND	ND	ND	ND
194	243E	ND	ND	ND	ND	ND	ND	ND	ND
195	243E	ND	ND	ND	ND	ND	ND	ND	ND
196	243D	ND	ND	ND	ND	ND	ND	ND	ND
197	243D	ND	ND	ND	ND	ND	ND	ND	ND
198	243D	ND	ND	ND	ND	ND	ND	ND	ND
199	243C	ND	ND	ND	ND	ND	ND	ND	ND
200	243C	ND	ND	ND	ND	ND	ND	ND	ND
201	243C	ND	ND	ND	ND	ND	ND	ND	ND
202	243B	ND	ND	ND	ND	ND	ND	ND	ND
203	243B	ND	ND	ND	ND	ND	ND	ND	ND
204	243B	ND	ND	ND	ND	ND	ND	ND	ND

Table 2 (Continued)

Sample No.	Building	TNR	RDX	TNT	NG	PETN	NC	Pb Sty	Tetrazene
205	243A	ND	ND	ND	ND	ND	ND	ND	ND
206	243A	ND	ND	ND	ND	ND	ND	ND	ND
207	243A	ND	ND	ND	ND	ND	ND	ND	ND
208	232H	ND	ND	ND	ND	ND	ND	ND	ND
209	232G	ND	ND	ND	+(2)	+	ND	ND	+(3)
210	232F	ND	ND	ND	ND	+	ND	ND	+(3)
211	232E	ND	ND	ND	ND	+	ND	ND	+(3)
212	232D	ND	ND	ND	ND	ND	ND	ND	ND
213	232C	ND	ND	ND	+	+	ND	ND	ND
214	232B	ND	ND	ND	ND	+	ND	ND	ND
215	232A	ND	ND	ND	ND	+	ND	ND	ND
400	222A	ND	ND	ND	ND	ND	ND	ND	ND
401	222A	ND	ND	ND	ND	ND	ND	ND	ND
402	222A	ND	ND	ND	ND	ND	ND	ND	ND
403	222A	ND	ND	ND	ND	ND	ND	ND	ND
404	222A	ND	ND	ND	ND	ND	ND	ND	ND
405	223A	ND	ND	ND	ND	ND	ND	ND	ND
406	223A	ND	ND	ND	ND	ND	ND	ND	ND
407	223B	ND	ND	ND	ND	ND	ND	ND	ND
408	223B	ND	ND	ND	ND	ND	ND	ND	ND
409	234	ND	ND	ND	ND	ND	ND	ND	ND
410	234	ND	ND	ND	ND	ND	ND	ND	ND
411	234	ND	ND	ND	ND	ND	ND	ND	ND
412	234	ND	ND	ND	ND	ND	ND	ND	ND
413	234	ND	ND	ND	ND	ND	ND	ND	ND
414	234	ND	ND	ND	ND	ND	ND	ND	ND
415	223A	ND	ND	ND	ND	ND	ND	ND	ND
416	223A	ND	ND	ND	ND	ND	ND	ND	ND
417	223A	ND	ND	ND	ND	ND	ND	ND	ND
418	223A	ND	ND	ND	ND	ND	ND	ND	ND
419	223A	ND	ND	ND	ND	ND	ND	ND	ND
420	223A	ND	ND	ND	ND	ND	ND	ND	ND
421	223A	ND	ND	ND	ND	ND	ND	ND	ND
422	223A	ND	ND	ND	ND	ND	ND	ND	ND
423	223B	ND	ND	ND	ND	ND	ND	ND	ND
424	223B	ND	ND	ND	ND	ND	ND	ND	ND
425	223B	ND	ND	ND	ND	ND	ND	ND	ND
426	223B	ND	ND	ND	ND	ND	ND	ND	ND
427	222A	ND	ND	ND	ND	ND	ND	ND	ND
428	222A	ND	ND	ND	ND	ND	ND	ND	ND
429	244	ND	ND	ND	ND	ND	ND	ND	ND
430	234	ND	ND	ND	ND	ND	ND	ND	ND

Table 2 (Continued)

Sample No.	Building	TNR	RDX	TNT	NG	PETN	NC	Pb Sty	Tetrazene
431	234	ND	ND	ND	ND	ND	ND	ND	ND
432	234	ND	ND	ND	ND	ND	ND	ND	ND
433	234	ND	ND	ND	ND	ND	ND	ND	ND
434	234	ND	ND	ND	ND	ND	ND	ND	ND
435	234	ND	ND	ND	ND	ND	ND	ND	ND
437	234	ND	ND	ND	ND	ND	ND	ND	ND
438	223A	ND	ND	ND	ND	ND	ND	ND	ND
454	234	ND	ND	ND	ND	ND	ND	ND	ND
455	234	ND	ND	ND	ND	ND	ND	ND	ND
456	234	ND	ND	ND	ND	ND	ND	ND	ND
457	223B	ND	ND	ND	ND	ND	ND	ND	ND
458	223A	ND	ND	ND	ND	ND	ND	ND	ND
459	223B	ND	ND	ND	ND	ND	ND	ND	ND
460	223B	ND	ND	ND	ND	ND	ND	ND	ND
461	223B	ND	ND	ND	ND	ND	ND	ND	ND
462	223B	ND	ND	ND	ND	ND	ND	ND	ND
463	223B	ND	ND	ND	ND	ND	ND	ND	ND
464	223A	ND	ND	ND	ND	ND	ND	ND	ND
466	223A	ND	ND	ND	ND	ND	ND	ND	ND
467	223A	ND	ND	ND	ND	ND	ND	ND	ND
468	223A	ND	ND	ND	ND	ND	ND	ND	ND
469	223B	ND	ND	ND	ND	ND	ND	ND	ND
470	223B	ND	ND	ND	ND	ND	ND	ND	ND
472	223B	ND	ND	ND	ND	ND	ND	ND	ND

- (1) ND = not detected.
- (2) TLC analysis gave a positive response. However, presence of interference(s) precludes positive identification and quantitation by TLC. HPLC analysis was used to confirm the presence of these materials at or about the TLC detection limit (50-75 ug/g for NG and PETN).
- (3) TLC analyses gave positive response. However, presence of interference(s) makes TLC identification and quantitation unreliable.

**Table 3. Hanley Area  
Positive Spot Spray Results (Buildings)**

<u>Building</u>	<u>Room</u>	<u>Spot Spray Number</u> *				
		<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>
218 A	101	+				
	102		+			
	104	+				
	105	+				
	106		+			
	108		+			
	109	+				
	112	+				
	113		+			
	121	+				
	124	+				
	127	+				
218 B	101		+			
	102		+			
	103		+			
	104		+			
	106				+	
	107		+			
	108				+	
	109		+			
	116		+			
	121		+			
	122	+				
	124	+				
	125		+			
	127		+			
	132		+			
	141			+		
	142		+			
	144		+			
	B02	+				
	B05	+				
218 C	102	+				
	104	+				
	106	+				
	108		+			
	111				+	
	115	+				

Table 3 (Continued)

Building	Room	<u>Spot Spray Number</u>				
		1	2	3	4	5
218 C	127		+			
	131		+			
	132		+			
	B02	+				
	B04	+				
	B05	+				
<hr/>						
219 A	102		+			
<hr/>						
219 D	101	+				
	103		+			
<hr/>						
219 G	103		+			
<hr/>						
220	101				+	(sill)
	101				+	(wall)
	101		+	(shelf)		
	102		+			
	105				+	
	106				+	
	107		+			

\*Compounds Identified by Spot Sprays

<u>Spot Spray Number</u>	<u>Compound(s) Detected</u>
1	Nitrocellulose, Nitroglycerine
2	PETN, HMX
3	DNT, TNT, TETRYL
4	Styphnic Acid, Lead Styphnate
5	Tetrayene

Table 4. Hanley Area  
Spot Spray Results (Magazines)

Magazine	Room	<u>Spot Spray Number</u>				
		1	2	3	4	5
219 E	101				+	
219 F	101		+			
219 H	101			+		
219 J	101		+			
227 A	101	+				
227 B	101	+				
227 J	101			+		
227 M	101			+		
227 O	101	+				
228 C	101	+				
228 F	101	+				
228 G	101		+			
228 N	101		+			
228 P	101		+			
228 W	101		+			
228 Y	101		+			
228 Y	B01	+				
229 K	101		+			
229 N	101		+			

Table 5. Hanley Area  
Physical Sample Results\*

Composite Sample #	Building	Analyte								Styphnic Acid	HMX
		2,4 DNT	2,6 DNT	TNT	TETRYL	PETN	NG	NC	Tetrazene		
S62434	218 A	ND	ND	ND	+	ND	ND	ND	ND	ND	<0.0075
S62447	218 B	ND	ND	ND	+	ND	ND	ND	ND	ND	3.8
S62468	218 C	ND	ND	ND	+	ND	ND	ND	ND	ND	<0.0075
S62497	219 A	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62424	219 C	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.0120
S62500	219 D	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62425	219 E	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62503	219 G	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62428	219 H	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.0120
S62481	220	ND	ND	ND	+	ND	ND	ND	ND	ND	<0.0075
S62490	227 (M)	ND	ND	ND	+	ND	ND	ND	ND	ND	2.1
S62493	227 (E)	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.029
S62420	228 (E)	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.0083
S62411	228 (M)	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62417	228 (W)	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62431	229	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62504	218 C (B03)	ND	ND	ND	ND	+	ND	ND	ND	ND	0.35
S62505	228 R (B01)	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075
S62506	228 Y (B01)	ND	ND	ND	ND	ND	ND	ND	ND	ND	<0.0075

\* All analyses were performed by TLC methodology except Tetrazene and HMX. Tetrazene analyses were performed by colorimetric spot test. HMX analyses were performed by quantitative HPLC procedure.

LEGEND: ND = Non-detectable  
(M) = Mid  
(E) = East  
(W) = West  
(+) = Positive (Detection Limit 1 µg/cm<sup>2</sup>)

**Table 6. Hanley Area  
Detection Limit, Precision, and Accuracy  
of Analytical Methods by Analyte**

Analyte	Detection Limit	Accuracy	Precision
Ag (in water)	0.5 ppb	0.85	0.10
Pb " "	10 "	0.89	0.10
Cr " "	5 "	0.92	0.10
Ni " "	10 "	1.03	0.10
Cd " "	0.5 "	0.93	0.10
Hg " "	0.4 "	0.87	0.10
Ag (on filter swipes)	1 $\mu\text{g}/\text{m}^2$		
Pb " " "	5 "		
Cr " " "	5 "		
Ni " " "	5 "		
Cd " " "	25 "		
Hg " " "	0.2 "		
Styphnic Acid (in water)	50 ppb	0.66	0.08
Tetrazene	500 "	1.01	0.03
HMX (in water)	8.4 "	0.96	0.041
NC (in water & soil)	9700 "	0.976	0.011
NG " " "	9700 "	0.976	0.011
PETN " " "	9700 "	0.976	0.011
TNT (in water)	1.2 "	0.60	0.35
Tetryl "	2.4 "	0.69	0.15
2,4-DNT "	0.5 "	0.83	0.14
2,6-DNT "	0.5 "	0.83	0.14
Explosives by TLC	1 $\mu\text{g}/\text{cm}^2$		
2,6-DNT by TLC	2.5 "		

**Table 7. Hanley Area  
Heavy Metal Analysis of Surface Swipes ( $\mu\text{g}/\text{M}^2$ )**

Bldg./ Magazine	Sample #	Pb	Ag	Ni	Hg	Cr	Cd
218 A	S62311	17300	4.3	61	<0.2	343	102
218 B	S62316	4920	8.6	74	<0.2	310	<25
218 C	S62322	5080	7	147	<0.2	449	<25
219 A	S62360	820	3.7	22	32	64	<25
219 B	S62295	3330	4.6	55	<0.2	126	<25
219 D	S62365	1920	8	64	<0.2	232	100
219 E	S62298	27200	24	30	<0.2	92	<25
219 G	S62370	1900	10	84	1.7	293	<25
219 J	S62301	4220	9.8	147	<0.2	364	28
220	S62329	5850	7.5	100	0.9	434	54
226 (W)	S62334	8140	3.1	34	<0.2	116	<25
227 (E)	S62351	4180	4.2	59	<0.2	170	<25
227 (M)	S62343	2870	3.2	33	<0.2	95	<25
227 T	S62285	800	<1	<5	<0.2	26	<25
228 (E)	S62268	4020	4.7	59	<0.2	300	<25
228 (M)	S62277	2870	3.9	53	<0.2	222	<25
228 (W)	S62286	3880	6.4	95	<0.2	515	40
229	S62304	2670	4.1	49	<0.2	328	<25
236	S62328	6180	5	40	<0.2	172	<25

**QUALITY CONTROL DATA\* ( $\mu\text{g}/\text{M}^2$ )**

Blank	-	<5(0)	<1(0)	<5(0)	<.2(0)	<5(0)	<1(0)
S.L. 1	-	6(2.5)	<1(.5)	<5(2.5)	.2(.2)	<5(2.5)	25(25)
S.L. 2	-	8(5)	1.1(1)	8(5)	.33(.4)	6(5)	50(50)
S.L. 3	-	12(10)	2(2)	14(10)	.79(.8)	11.5(10)	100(100)
S.L. 4	-	27(25)	5.2(5)	27(25)	2.06(2)	26.5(25)	246(250)
S.L. 5	-	53(50)	10.2(10)	55(50)	4.16(4)	56(50)	520(500)

\* Expected value in parenthesis

LEGEND: S.L. = Spike Level

Table 8. Hanley Area  
Powder Well Sample Results (ppb)

Composite Sample	Location	2,4 DNT	2,6 DNT	TNT	Tetryl	PETN	NG	NC	Tetrazene	Styphnic Acid	HMX
S62407	PW425	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62375	PW0218A	<0.5	<0.5	<1.2	4.0	<9700	<9700	<9700	< 500	<50	<8.4
S62380	PW0218B	<0.5	<0.5	<1.2	4.6	<9700	<9700	<9700	< 500	<50	<8.4
S62384	PW0218C	<0.5	<0.5	<1.5	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62399	PW226 (W)	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62389	PW227 (E)	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62394	PW227 (M)	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62404	PW228 (E)	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62408	PW228 (W)	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4

QUALITY CONTROL DATA\* (ppb)

S63109	Blank	NA	NA	NA	NA	NA	NA	NA	NA	NA	<8.4(0)
S63102	Blank	NA	NA	NA	NA	NA	NA	NA	NA	NA	<8.4(0)
S63099	Spike Level	NA	NA	NA	NA	NA	NA	NA	NA	NA	39(40)
S63100	Spike Level	NA	NA	NA	NA	NA	NA	NA	NA	NA	75(80)
S62514	Duplicate*	NA	NA	NA	NA	NA	NA	NA	< 500(<500	NA	NA
S62515	Spike	NA	NA	NA	NA	NA	NA	NA	2.5(2.5)	NA	NA
S62375	Spike	NA	NA	NA	NA	NA	NA (1.6x10 <sup>4</sup> )	NA (10 <sup>4</sup> )	NA	131(200)	NA
S62407	Spike	3.3(5)	3.3(5)	4.5(5)	7.3(5)	NA	NA	NA	NA	NA	NA
S62541	DW Spike	<0.5(0)	<0.5(0)	<0.5	<0.5(0)	NA	NA	NA	NA	NA	NA
S62542	DW Spike	0.42 (0.5)	0.5 (10.5)	0.58 (0.5)	1.5 (0.5)	NA	NA	NA	NA	NA	NA
S62543	DW Spike	0.75(1)	0.80(1)	1.4(1)	2.6(1)	NA	NA	NA	NA	NA	NA
S62544	DW Spike	2.0(2)	2.1(2)	3.5(2)	3.5(2)	NA	NA	NA	NA	NA	NA
S62545	DW Spike	5.1(5)	4.3(5)	6.1(5)	6.6(5)	NA	NA	NA	NA	NA	NA
S62546	DW Spike	8.8(10)	8.8(10)	11.9(10)	11.6(10)	NA	NA	NA	NA	NA	NA

\* Expected value in parenthesis

Legend: NA = Not analyzed  
#D.W. Spike = Distilled water spike

Table 9. Hanley Area  
Sewer Sample Explosive Results (ug/l)

Sample	Sewer Location	2,4 DNT	2,6 DNT	TNT	Tetryl	PETN	NC	NC	Tetrazene	Styphnic Acid	HMX
S62512	428	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62513	430	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62514	432	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62515	434	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4
S62516	436	<0.5	<0.5	<1.2	<2.4	<9700	<9700	<9700	< 500	<50	<8.4

QUALITY CONTROL DATA\* (ppb)

S63109	Blank	NA	NA	NA	NA	NA	NA	NA	NA	NA	<8.4(0)
S63102	Blank	NA	NA	NA	NA	NA	NA	NA	NA	NA	<8.4(0)
S63099	Spike Level	NA	NA	NA	NA	NA	NA	NA	NA	NA	39(40)
S63100	Spike Level	NA	NA	NA	NA	NA	NA	NA	NA	NA	75(80)
S62514	Duplicate*	NA	NA	NA	NA	NA	NA	NA	<500(<500)	NA	NA
S62515	Spike	NA	NA	NA	NA	NA	NA	NA	2.5(2.5)	NA	NA
S62375	Spike	NA	NA	NA	NA	NA	NA	NA	(1.6x10 <sup>4</sup> )(10 <sup>4</sup> )	131(200)	NA
S62407	Spike	3.3(5)	3.3(5)	4.5(5)	7.3(5)	NA	NA	NA	NA	NA	NA
S62541	DW Spike	<0.5(0)	<0.5(0)	<0.5	<0.5(0)	NA	NA	NA	NA	NA	NA
S62542	DW Spike	0.42 (0.5)	0.5 (10.5)	0.58 (0.5)	1.5 (0.5)	NA	NA	NA	NA	NA	NA
S62543	DW Spike	0.75(1)	0.80(1)	1.4(1)	2.6(1)	NA	NA	NA	NA	NA	NA
S62544	DW Spike	2.0(2)	2.1(2)	3.5(2)	3.5(2)	NA	NA	NA	NA	NA	NA
S62545	DW Spike	5.1(5)	4.3(5)	6.1(5)	6.6(5)	NA	NA	NA	NA	NA	NA
S62546	DW Spike	8.8(10)	8.8(10)	11.9(10)	11.6(10)	NA	NA	NA	NA	NA	NA

\* Expected value in parenthesis

Legend: NA = Not analyzed  
#D.W. Spike = Distilled water spike

**Table 10. Hanley Area  
Sewer Sample Heavy Metal Results (ug/l)**

Sample #	Sewer #	Pb	Ag	Ni	Hg	Cr	Cd
S62507	428	230	<0.5	14	<0.4	<5.0	0.9
S62508	430	18	<0.5	115	<0.4	<5.0	<0.5
S62509	432	270	<0.5	14	<0.4	<5.0	1.2
S62510	434	190	<0.5	<10	<0.4	<5.0	3.3
S62511	436	<10	<0.5	<10	<0.4	<5.0	<0.5

**QUALITY CONTROL DATA\* (ppb)**

S62507	Dupli.	220(230)	<.5(.5)	17(14)	<.4(.4)	<5(5)	1.1(.9)
S62510	Spike	330(340)	8.4(10)	49(50)	3.2(4)	96(100)	112(133)
Blank	-	<10(0)	<.5(0)	<10(0)	<.4(0)	<5(0)	<.5(0)
S.L. 1	-	17(5)	<.5(.5)	10(5)	<.4(.2)	5(5)	.7(.5)
S.L. 2	-	10(10)	.8(1)	8(10)	.4(.4)	10(10)	.9(1)
S.L. 3	-	19(20)	1.6(2)	**-(20)	.84(.8)	18(20)	1.9(2)
S.L. 4	-	45(50)	4.2(5)	55(50)	1.96(2)	47(50)	4.7(5)
S.L. 5	-	94(100)	8.5(10)	103(100)	3.36(4)	92(100)	9.4(10)

\* Expected values in parentheses

\*\* Sample lost in processing

**APPENDIX E**

**ANALYTICAL METHODS AND QUALITY ASSURANCE DATA FOR THE**

**GOODFELLOW US ARMY RESERVE CENTER SURVEY**

## 1. Analytical Methods

a. Spot Spray Test Methods. Preliminary evaluation of the presence of selected explosives was conducted by spraying building surfaces with reagents which give positive color responses in the presence of certain explosive materials. These tests were conducted using the reagents listed in Table 1. The test for RDX, etc. was performed using two sprays in succession. The first spray reduced the nitro groups in these explosives to nitrite while the second spray gave a positive response to the liberated nitrite. The second of these sprays was also used to obtain a measure of any background nitrite containing material which would contribute to a flow positive response to this test. Each spray was applied to different spots within a one-meter-square area, with a suitable interval between sprays to prevent mixing of spray materials on the building surface.

Table 1. Spray Tests for Selected Explosives

<u>Munition</u>	<u>Indicating Reagent</u>	<u>Detection Limit<sub>2</sub> µg/cm<sup>2</sup></u>
TNT	A mixture of 1 percent 1,3 diphenylacetone plus 0.2 percent tetramethylammonium hydroxide in ethanol	0.4
RDX, NC, NG, PETN	(1) 10% Zinc dust in toluene (2) 0.35% each of procaine + N,N <sub>2</sub> -dimethylnaphthylamine in 50% acetic acid	0.4
TNR,PbSty*	A mixture of 10% SnCl <sub>2</sub> ·2H <sub>2</sub> O in 5% NaOH (filtered)	0.4
Tetrazene	The reaction products from 10% NaOH, 10% Na <sub>2</sub> [Fe(NO)(CN <sub>5</sub> )]·2H <sub>2</sub> O and 10% K <sub>3</sub> FeCN <sub>6</sub> , mixed with an equal volume of acetone.	0.4

\*PbSty = lead styphnate

b. Thin Layer Chromatography Methods. The analyses were conducted using Silica Gel plates (LK5DF) with a 15 percent butyl acetate in petroleum ether (bp 30-60C) developer and a series of visualization techniques. A list of the R<sub>f</sub> values and visualization procedures is given in Table 2.

**Table 2. TLC Parameters for Selected Explosives**

<u>Munition</u>	<u>R<sub>f</sub></u>	<u>Visualization Procedure</u>	<u>Detection Limit, <math>\mu\text{g/gm}</math></u>
TNT	0.92	Spray with 10% KOH in methanol	25
TNR, PbSty	0.0	Visualize in daylight and $\mu\text{v}$	25
NC	0.0	Spray with modified Greiss reagent	50
NG	0.76	after heating at 105°C for 20	
PETN	0.90	minutes.	
RDX	0.09	Same as above after visualizing in $\mu\text{v}$ light	50

All physical samples were weighed in 1.0 gram aliquots where sample size permitted and mixed in a sonic bath for 0.5 hour with 2 ml acetone. A 50  $\mu\text{l}$  aliquot of this extract was then spotted on the TLC plate, the solvent allowed to evaporate, and the plate developed in a saturated tank for at least 15 min. The plate was then removed from the tank, air dried, and visualized sequentially according to the tests in Table 2. Quantitation was accomplished by comparison with known standards.

The determination of tetrazene was accomplished using a separate procedure. Because of the low organic solubility, it was not possible to operate sufficient loadings on the TLC plate to permit visualization and identification. Therefore, a swabbing procedure was employed in which the sample, acidified with 0.01  $\text{NHCl}$  (to solubilize tetrazene), was allowed to soak into the Q-tip. The Q-tip was then sprayed with the appropriate visualization spray (see Table 1). This test was found to be very selective and sensitive for tetrazene in the presence of the other explosives.

c. HPLC Analysis. All high performance liquid chromatographic (HPLC) analyses were performed using Waters Model 6000A Solvent Delivery System a LDC UNIII Monitor, a DuPont Model 834 Automatic Sampler, and a Hewlett Packard 3385 data system.

Water Sample Preparation and Analysis. Water samples obtained during this study were stored at 4°C in amber-colored bottles fitted with aluminum foil-lined lids. Prior to preparation for analysis, each sample was allowed to warm to ambient temperature. These samples were then filtered in 50 ml aliquot, the residue rinsed with 1 ml acetone and buffered with 1.0 ml (pH = 7) phosphate buffer. A 5 ml aliquot was then withdrawn, spiked with 50  $\mu\text{l}$  of 10.0 ppt m-nitrophenol in acetone (IS), and placed in a screw cap vial for subsequent HPLC analysis. Chromatographic conditions suitable for the separation and quantitation of TNR, RDX, TNT, NG, and PETN are described in Table 3.

**Table 3. Operating Parameters for HPLC Analyses**

Column	C <sub>18</sub> reversed phase (Waters Radial System)	Compression Separation
Mobile Phase	40% CH <sub>3</sub> CN/60% H <sub>2</sub> O	50% CH <sub>3</sub> CN/60% H <sub>2</sub> O
Flow Rate	1.5 ml/min	1.5 ml/min
Detection Wavelength	254 nm	254 nm
Attenuation	0.128 AUFS	0.016 AUFS
Injection Volume	20 µl	20 µl
Column Pressure	1000 psig	900 psig
Column Temperature	Ambient	Ambient
N <sub>eff</sub>	5000-7000 plates	5000-7000 plates

## **2. Quality Assurance**

### **a. Analysis for Heavy Metals**

#### **(1) Lead Chromium and Cadmium**

The quality assurance aspects of the inductively coupled plasma spectroscopy method used to determine lead, chromium, and cadmium were evaluated by determining the percent recovery of spiked samples. Recoveries from spiked acid swipe samples were Pb-83%, Cr-88%, and Cd-108%.

#### **(2) Mercury**

Quality assurance of the atomic adsorption method used for mercury analyses was also evaluated by determining recoveries from spiked samples. A sample was spiked with mercury and carried through the analytical procedure. Recovery from spiked mercury samples was 71 percent. Previous data is presented below.

#### **(a) Precision Data for Lead, Chromium and Cadmium**

<u>Spike Mass (Micrograms)</u>	<u>Relative Standard Deviation of Measured Mass (s/x)</u>		
	<u>Chromium</u>	<u>Cadmium</u>	<u>Lead</u>
10	.113	.031	.107
20	.049	.063	.076
200	.069	.050	.053
1000	.082	.043	.043

(b) Precision Data for Mercury

<u>Spike Mass (Micrograms)</u>	<u>Relative Standard Deviation of Measured Mass (s/<math>\bar{x}</math>)</u>
0.2	.104
0.4	.063
1.0	.056
3.0	.069

b. Analysis for Organics

(1) Quality Assurance Procedures for Spot Sprays. Each spray test series included a test for background nitrite, which may give false positive response for the NG, PETN, NC, and RDX tests. As no "blank" surface was apparent, no other "blank" samples could be run for these tests. However, at least once each day, the responsiveness of each spray was checked by spraying swabs which had been dipped in 0.012 percent solutions of the respective explosives. Such procedure constituted an analysis of "standard" samples.

(2) Quality Assurance Procedures for TLC Analyses. During the TLC analyses for the selected explosives, a series of blanks, standards, and spiked samples were routinely analyzed. Blank samples consisted of 2 ml acetone. Standard samples consisted of spiking known amounts of each explosive as a mixture onto the TLC plate. Also, a series of spiked samples were generated using a physical sample which had been analyzed previously and found to be free of munitions. Four one-gram aliquots were taken, spiked successively at the 25 ug/gm, 100 ug/gm, 500 ug/gm, and 0 ug/gm level, and analyzed by the procedures described above. The results of these analyses confirmed the validity of our procedures for determining each compound in actual physical samples from SLOP.

(3) Quality Assurance Procedures for HPLC Analysis.

(a) Sample Integrity. All water samples were collected in glass jars equipped with tight-fitting, aluminum-foil-lined lids. All samples were stored at 4°C in separate facility from the analytical lab prior to their preparation for analysis.

(b) Instrument Calibration. The calibration of the LDC UWIII Monitor consisted of following manufacturer's recommended procedures for evaluation of short-term drift and noise. The detection cell was cleaned and rinsed with organic solvents and all mirrors and lenses realigned and cleaned or replaced as appropriate. The instrument was allowed to stabilize for 1 hour with an N<sub>2</sub> purge through the detector cell, and the drift and noise was evaluated. Output noise at 254 nm was less than  $3 \times 10^{-4}$  AU, with an attenuation  $\times 0.0001$  AuFS, as per specifications. Drift was less than 0.01 A per 24 hours under these conditions.

(c) Material. All organic solvents used in these analyses were distilled-in-glass grade obtained from Burdick and Jackson Laboratories, Muskegon, Michigan. Table 4 describes the authentic explosive standards used in this study and the sources from which they were obtained. TNT, RDX, and TNR were prepared as 5.0 ppt solutions in acetone while NG and PETN were prepared as their 20.0 ppt acetone solution. These stock solutions were stored at 4C throughout the study. As fresh

standard solution of these explosives were required, they were prepared from these stored stock solutions. Distilled water was used to prepare standard solutions and the chromatographic mobile phase for HPLC analysis. This water was obtained from a Millipore/Milli-Q water purification system.

Table 4. Sources of Explosives Reference Materials

<u>Compound</u>	<u>Source</u>
2,4,6-Trinitrotoluene (TNT)	Edgewood Arsenal, Edgewood, MD
2,4-Dihydroxy-1,2,5-trinitrobenzene (TNR)	Eastman Organic Chemicals, Rochester, NY
1,3,5-Trinitro-1,3,5-triazocyclohexane (RDX)	Edgewood Arsenal, Edgewood, MD
Pentaerythritol tetranitrate (PETN)	Sandia Laboratories, Albuquerque, NM
Trinitroglycerin (NG)	ICI American Inc., Wilmington, DE
m-Nitrophenol	Eastman Organic Chemicals, Rochester, NY
Tetrazene	Olin Corp., East Altan, IL
Lead Styphnate (normal) (PbSty)	Olin Corp., East Altan, IL

These stock solutions were examined by HPLC chromatography for the presence of impurities, by injecting 20 µg of TNT, TNR, and RDX and 100 µg of PETN and NG. In this manner an impurity present at or above the 1 percent level would have been observed assuming any such material had a uv extinction coefficient similar to that of the parent compound. No single such species was observed although several such materials may have been present in trace amounts and whose total percentage in the standard solution could not be determined using these techniques.

Additionally, nuclear magnetic resonance (NMR) and infrared spectroscopy (IR) were used to evaluate these materials for the presence of impurities. These examinations were conducted on 10 mg portions of each standard such that a 1 percent impurity would be present at the 100 µg level. However, such an examination is not suitable for determining any impurities which have similar resonating or absorbing frequencies or extremely small "response factors."

(d) Standard Curves. Standard curves were constructed from solutions of six different concentrations of each explosive in 100 µg/ml m-nitrophenol solution. Each explosive was prepared as a stock solution in acetone from which the standard solutions were generated by combining appropriate amounts of these stock solutions in a single vial. The analytes were divided into two groups corresponding to their common methods of HPLC analysis (i.e., TNT, TNR, and RDX, and NG and PETN). Hence, six separate solutions were prepared containing TNR, RDX, and TNT at each of the six concentrations. Appropriate amounts of each stock solution were combined in a single vessel with 500 µg of m-nitrophenol and the mixture diluted to 5 ml with water. Similarly, PETN and NG standard solutions were prepared in 3 ml acetone containing 500 µg of m-nitrophenol and diluted to 5 ml with water. Each of the above six concentrations were replicated four times.

Standard curves were generated by single injection of each of the 24 vials obtained from the above procedure. Detection limits were determined from such curves using the methods of Hubaux and Vos. The instrumental detection limit for TNR, RDX, and TNT was selected\* at 10 µg/ml and at 40 µg/ml for PETN and NG.

(e) Accuracy and Precision. Once each analytical method was selected, the accuracy of these methods was evaluated in the following manner. Spiked water samples were analyzed in replicates of four at each of several concentrations based on the instrumental detection limit for each analyte. These spiked samples were prepared at 1/2 DL, DL, 2DL, 5DL--to the upper limit of reportable data for each method. DL for water samples was taken as the instrumental detection limit since these samples required essentially no dilutions or concentration prior to analysis. For a complete description of the sample preparation and analysis procedures, see the preceding sections of this report.

The accuracy and precision of the HPLC analysis method was determined at the detection limits of each compound where:

$$\text{Inaccuracy} = \frac{\bar{x} - \text{target}}{\text{target}} = (\text{fraction recovered}) - 100$$

and

$$\text{Imprecision} = \frac{S}{\bar{x}} .$$

The inaccuracy for TNR, RDX, and TNT was less than 0.50 and the imprecision was greater than 0.20 percent at 10 ppm. These values were less than 0.90 and 0.30 for NG and PETN at 40 ppm. The accuracy and precision improved dramatically at concentrations above the detection limits. However, since all samples analyzed contained analyte levels below the detection limit, only the above values are reported for these analyses.

(f) Blanks, Spiked Blanks, and Standard Solutions. Once the accuracy and precision of each of these analytical methods were verified, the continued performance of these sample preparation and analysis procedures was monitored by examining blank and spiked blank samples. Such samples were prepared and analyzed with every group of 10 or less water and soil samples. Blank water samples were generated in the laboratory and consisted of aliquots of distilled water. Blank water samples were spiked by injecting 50 ml blank water with either 50 µl each of 5.0 mg/ml solutions of TNR, RDX, and TNT or with 100 µl each of 20 mg/ml solutions of PETN and NG.

The validity of the standard curves determined for each explosive was monitored regularly by analyzing standard solutions with every set of samples analyzed. The concentration of these standard solutions was determined and if these concentrations deviated by more than 10 percent of their known values, new standard curves were constructed.

---

\* Actual method values were slightly less than these values and varied between compounds. A round number to represent detection limits was selected for convenience.

(g) Evaluation of Auto-Sampler. An evaluation was made of the reproducibility of the injections made by the auto-sampler used in these analyses. Seven LC vials were filled from a solution containing 100 µg/ml of TNT and 100 µg/ml IS. These vials were then analyzed with 3 injections per vial to yield a total of 21 injections of this solution. The peak areas of each explosive were then measured and the average and standard deviation obtained. However, because of the known poor reproducibility of injections in HPLC, the method of internal standards was chosen for quantitation of all analytes in this study. Consequently, the peak area ratio of analyte to IS was also determined for these 21 injections and the corresponding average and standard deviation calculated. Similarly, a 400 µg/ml solution of NG was evaluated. The results of these evaluations are given in Table 5, where it is seen that good reproducibility (i.e., precision which equals  $S/\bar{x}$ ) is achieved when peak area ratios are determined.

Table 5. Reproducibility of Automatic Injector

<u>Analyte</u>	<u>Avg. Peak Area</u>	<u>S</u>	<u>S/<math>\bar{x}</math></u>	<u>Avg. Peak Area Ratio</u>	<u>S</u>	<u>S/<math>\bar{x}</math></u>
TNR	54,990	8742.3	0.16	0.50836	0.0153	0.03
RDX	83,657	11844.0	0.14	0.73955	0.0161	0.02
TNT	155,200	24016.0	0.16	1.43610	0.0380	0.03
NG	15,612	1394.9	0.09	0.12490	0.0112	0.09

**APPENDIX F**

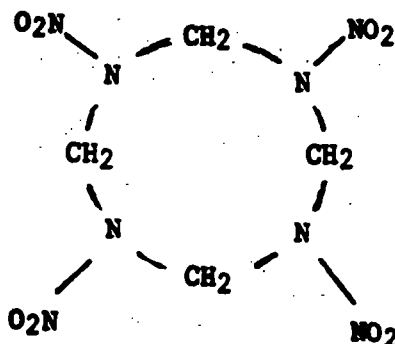
**ANALYTICAL METHODS AND QUALITY ASSURANCE DATA FOR THE HANLEY AREA SURVEY**

DETERMINATION OF HMX ON BUILDING SURFACES

- I. Application: This method is used to semiquantitatively determine the concentration of HMX (1,3,5,7-tetranitro-1,3,5,7-tetracyclo-octane) on building surfaces. It is primarily designed as a screening technique, not a quantitative method.
- A. Tested Concentration Range - The concentration range analyzed is 0.006  $\mu\text{g}/\text{cm}^2$  to 0.15  $\mu\text{g}/\text{cm}^2$  on building surface.
- B. Sensitivity Limits - The signal to noise ratio was 11:1 for 2 ng injected of HMX.  
At the Detection limit,  
1 ng of HMX =  $1.4 \times 10^{-4}$  AU
- C. Detection Limits - Employing the method of Hubeaux and Vos, the detection limit for HMX in water was determined to be 8.0  $\mu\text{g}/\ell$  (lowest level std.) which correspond to 0.0063  $\mu\text{g}/\text{cm}^2$ .
- D. Interferences - Components which elute with the same retention time as HMX and absorb UV light (254 nm) will interfere.
- E. Analysis Rate - Approximately twenty samples can be analyzed by a worker in an 8 hour day after instrument calibration.

II. Chemistry

Potential explosive, toxic inhalation and skin absorption hazards exist. Chemical structure for HMX:



Alternate nomenclature - cyclotetramethylene tetranitramine; 1,3,5,7-tetranitrol-1,3,5,7-tetracyclooctane; homocyclonite

CAS RN # - 479-45-8

Melting point - 276-7°C

Boiling point - 337°C (explodes)

### III. APPARATUS

- A. Instrumentation - An Altex Model 100A pump equipped with an Altex Model 420 microprocessor. An LDC UV III Monitor (1203) detector, a Rheodyne Model 7120 injection valve with a 100  $\mu$ l loop, and a Varian Model 9176 recorder. A Spectra Physics SP 4000 data system.
- B. HPLC Parameters -
1. Column: Spherisorb 5 micron ODS, 250x4.6 mm.
  2. Mobile Phase: Acetonitrile/water, 40/60, v/v.
  3. Flow: 1 ml/min.
  4. Temperature: 25°C.
  5. Detector wavelength and sensitivity: 254 nm, 0.004 AUFS.
  6. Recorder Span: 10 mv/full scale.
  7. Injection volume: 100  $\mu$ l.
  8. Retention time for HMX: 9.8 minutes.
- C. Hardware/Glassware -
1. Microspatula.
  2. 5, 50, 100, 250 ml volumetric flasks.
  3. 4 ounce bottles with Teflon lined screw caps.
  4. 2 dram vials with Teflon lined screw caps.
  5. Pasteur pipets (5 3/4") and bulbs.
  6. 5 ml pipet and bulb.
  7. Millipore solvent filtering apparatus.
  8. Millipore filters, 0.45 $\mu$ , type HA, 47 mm.
  9. 100  $\mu$ l Hamilton syringe.
  10. Refrigerator and freezer.
  11. Cotton swabs (Q-tips).
- D. Chemicals -
1. Acetone, DIG grade, Burdick and Jackson.
  2. Millipore, Milli-Q purified water.

### IV. STANDARDS

#### A. Calibration Standards -

Stock. Ten milligrams of HMX are transferred to a 50 ml volumetric flask. The flask is diluted to the mark with acetone and mixed well.

### V. PROCEDURE

- A. Sample Collection and Preparation - Place 1.5 milliliters of acetone in a 2 dram vial. Mark a one meter square area on the surface to be sampled. Using cotton swabs (Q-tips) wetted with

acetone to swab five 20 square centimeter areas within the marked area. Place the swabs in the vial, cap tightly, and return to the laboratory for analysis.

Place 100  $\mu$ l of the acetone extract in a five milliliter volumetric flask and dilute to volume with distilled water. Mix well, place in a screw capped 2-dram vial and store refrigerated until analyzed.

- B. Calibration - Inject 100  $\mu$ l of each standard two times onto the chromatographic system. A solvent blank, represented by distilled water, is also analyzed. Record the concentration, retention time and peak height for HMX. Average the peak heights for the duplicate injections of each standard.
- C. Analysis - Inject 100  $\mu$ l of each diluted sample onto the chromatographic system. Record the retention time and peak height for peaks eluting at the same time as HMX (within 0.1 minute of the HMX standard).

#### VI. CALCULATIONS

A linear regression curve (concentration vs. peak height) is generated from the HMX calibration data. The diluted sample concentrations are interpolated using the curve.

The relationship between diluted extract concentration and concentration on building surface is:

$$\mu\text{g}/\text{cm}^2 = \frac{\mu\text{g}/\text{l in diluted extract}}{1330}$$

#### VII. REFERENCE

1. Battelle method "Determination of HMX in Water".

##### Spiked Recovery Data for Water (from reference 1)

##### HMX in Water

<u>N</u>	<u>Target Value ng/ml</u>	<u>Found Value ng/ml</u>			
4	8.0	7.5	7.5	7.5	7.5
4	20	19	19	20	20
4	40	41	39	47	37
4	80	79	76	80	76
4	200	193	193	191	191

DETERMINATION OF Hg, Cd, Ag, Ni, Cr AND Pb  
BY ATOMIC ABSORPTION SPECTROMETRY

**I. Application**

The method is used for the determination of Hg, Cd, Ag, Ni, Cr, and Pb in water and on filter papers.

**A. Tested Concentration Range:**

The concentration range for each metal is:

	<u>In Solutions (ppb)</u>	<u>In Filter Mixes (<math>\mu\text{g}/\text{m}^2</math>)</u>
Hg	0.2 - 4.0	0.2 - 4.0
Cd	0.5 - 10.0	25 - 500
Ag	0.5 - 10.0	0.5 - 10.0
Ni	5.0 - 100	2.5 - 50
Cr	5.0 - 100	2.5 - 50
Pb	5.0 - 100	2.5 - 50

**B. Sensitivities: N/A**

C. Detection Limits:

	<u>In Solutions (<math>\mu\text{g}/\text{l}</math>)</u>	<u>In Filters (<math>\mu\text{g}/\text{m}^2</math>)</u>
Hg	0.90	0.20 (lowest std)
Cd	0.54	27.
Ag	0.50 (lowest std)	1.1
Ni	17.	10.
Cr	5.0 (lowest std)	6.1
Pb	7.2	6.3

- D. Interferences: Possible interference of sulfide or sulfite with the Hg determination avoided by oxidation of the samples and the use of a silver wool collector. The use of a background corrector for the determination of the other metals is effective for most interferences.

E. Analysis Rate:

Flame Excitation--35 determinations per day including sample preparation.

Graphite Furnace Excitation--25 determinations per day including sample preparation.

Mercury--15 determinations per day including sample preparation.

II. Chemistry

In direct aspiration atomic absorption spectroscopy a sample solution containing the metals in ionic form is aspirated and atomized in a flame. Free unexcited ground state atoms are formed in the flame and absorb light energy from a hollow cathode lamp whose cathode is made of the element being determined. Since the wavelength of the light beam is characteristic of only the metal being determined, the light energy absorbed is a measure of the concentration of that metal in the sample.

The principle involved in the furnace technique is essentially the same as with direct aspiration except a furnace, rather than a flame, is used to atomize the sample. As a greater percentage of available analyte atoms are vaporized and dissociated for absorption in the tube than in the flame, the use of small volumes or detection of low concentrations of elements is possible.

To determine mercury all forms of mercury are converted to mercuric ions by proper digestion. The mercuric ions in the sample solution are reduced to the elemental state, purged from the solution and passed through an optical absorption cell positioned in the light path of an atomic absorption spectrometer. The absorption of radiation at 253.7 nm from a mercury vapor lamp is measured, providing a measure of mercury concentration in the sample.

### III. Apparatus

#### A. Instrumentation:

##### Mercury

- Perkin-Elmer Model 305B atomic absorption spectrophotometer equipped with a mercury hollow cathode lamp.
- Absorption cell. A cylindrical tube with quartz windows approximately 25 mm ID and 150 mm long with an inlet and outlet sidearms, the inlet having a 12/5 female ball joint. The cell is mounted in the optical path of the atomic absorption spectrophotometer.
- Collector tubes constructed of 7 mm Pyrex tubing, 10 cm long with 12/5 male and female ball joints on each end, respectively. Each is wound with 100 cm of 22-gage chromel A wire and loaded with 0.7 gram of silver wool.
- Mercury reduction apparatus. The reaction vessel is a 150 ml gas-washing bottle with a fritted glass bubbler tube. The outlet tube is fitted with a 12/5 male ball joint. Nitrogen is used as the purging gas and it flows through an activated charcoal trap, a flowmeter, and on into the inlet tube of the reaction vessel. A by-pass circuit for the nitrogen is provided to by-pass the reaction vessel after the mercury has been released from solution and collected on the silver wool.
- Strip-chart recorder.
- Variac controller - 5 amp.
- Compressed nitrogen gas.

Cadmium, Silver, Nickel, Chromium, and Lead

- Perkin-Elmer Model 5000 atomic absorption spectrophotometer equipped with the proper hollow cathode lamp, background corrector, automated sample injector, HGA 500 graphite furnace and stripchart recorder.

B. Atomic Absorption Parameters:

	<u>Hg</u>	<u>Cd</u>	<u>Ag</u>	<u>Ni</u>	<u>Cr</u>	<u>Pb</u>
Wavelength (nm)	253.7	228.8	328.1	232.0	357.9	283.3
Slit Width (nm)	0.7	0.7	0.7	0.2	0.7	0.7
Light Source	← Hollow Cathode Lamps →					
Excitation	--	A/A	A/A	A/A	N/A	A/A

A/A = Air/acetylene

N/A = Nitrous oxide/acetylene

HGA Excitation Parameters

	<u>Cd</u>	<u>Ag</u>	<u>Ni</u>	<u>Cr</u>	<u>Pb</u>
Drying Temp °C	100	100	100	100	100
Char Temp. °C	400	400	1000	1100	700
Atomization Temp. °C	2200	2600	2700	2700	2300
Internal Flow (ml/min)	80	80	40	200	100

C. Hardware/Glassware

500-ml beakers with cover glasses  
 250-ml beakers with cover glasses  
 250-ml Erlenmeyer flasks with cover glasses  
 10-ml volumetric flasks  
 25-ml volumetric flasks  
 100-ml volumetric flasks  
 1-liter volumetric flasks  
 Eppendorf pipets  
 Glass pipets

D. Chemicals:

- Sulfuric acid, concentrated--reagent grade.
- Perchloric acid (60 percent)--reagent grade.
- Nitric acid, concentrated--special reagent grade of low Hg content.
- Hydrochloric acid, concentrated--reagent grade.
- Stannous Chloride--dissolve 20 g of  $\text{SnCl}_2 \cdot 2 \text{H}_2\text{O}$  in 20 ml of concentrated HCl and dilute to 100 ml with deionized water.
- Hydroxylamine Hydrochloride (10 percent)--dissolve 10 g of hydroxylamine hydrochloride in water and dilute to 100 ml.
- Potassium Permanganate (5 percent)--dissolve 50 g of  $\text{KMnO}_4$  in water and dilute to 1 liter.
- Potassium Persulfate (5 percent)--dissolve 5 g of potassium persulfate in 100 ml of water.
- Stock Solutions--Fisher Scientific Certified Standards--1000  $\mu\text{g/ml}$  for each metal.
- Iodine Monochloride Solution (1 M Stock)--add 444 ml concentrated HCl to 444 ml of a 25 percent (W/v) potassium iodide solution, cool and add 75 g of potassium iodate with stirring. Cool and dilute to 1 liter with water.
- Iodine Monochloride Solution (0.1 M)--dilute 100 ml of the stock solution to 1 liter with water and mix well.
- Sodium Hydroxide Solution (20 percent)--dissolve 20 g of NaOH in 100 ml water.
- Hydrogen Peroxide (30 percent)--reagent grade.

E. Reagents: None

IV. Standards

A. Calibration Standards:

1. Stock. Fisher Scientific Certified standard solutions containing 1000  $\mu\text{g}$  metal/ml.

## 2. Working:

10 ppm solution - 1.0 ml of stock solution diluted to 100 ml.

1.0 ppm solution - 10 ml of 10 ppm solution diluted to 100 ml.

0.1 ppm solution - 10 ml of 1.0 ppm solution diluted to 100 ml.

Blank - 5 ml conc.  $\text{HNO}_3$  diluted to 100 ml.

### B. Control Spikes:

Spiked solutions of Pb, Ni, and Cr were prepared by adding 0.05, 0.1, 0.2, 0.5 and 1.0 ml of their 10 ppm solutions to 100 ml of water to provide concentrations of 5, 10, 20, 50, and 100 ppb. Spiked solutions of Ag and Cd were prepared by adding 0.05, 0.1, 0.2, 0.5 and 1.0 ml of their 1.0 ppm solutions to 100 ml to provide concentrations of 0.5, 1.0, 2.0, 5.0, and 10 ppb. Spiked solutions of Hg were prepared by adding 0.05, 0.1, 0.2, 0.5, and 1.0 ml of the 0.1 ppm Hg solution to 25 ml providing concentrations of 0.2, 0.4, 0.8, 2.0 and 4.0 ppb.

Spiked filter samples of Pb, Ni, and Cr were prepared by adding 0.25, 0.5, 1.0, 2.5 and 5.0 ml of the 10 ppm solutions to a filter paper to provide concentrations of 2.5, 5.0, 10, 25, and 50  $\mu\text{g/square meter}$ . Spiked filter samples of Ag were prepared by adding 0.5, 1.0, 2.0, 5.0, and 10.0 ml of the 1 ppm solution to a filter paper to provide concentration of 0.5, 1.0, 2.0, 5.0, and 10.0  $\mu\text{g/square meter}$ . Concentration of 25, 50, 100, 250, and 500  $\mu\text{g Cd/square meter}$  were obtained by adding 2.5, 5.0, 10.0, 25.0, and 50.0 ml of the 10 ppm standard solution to a filter paper. Mercury standards of 0.2, 0.4, 0.8, 2.0, and 4.0  $\mu\text{g/square meter}$  were prepared by adding 1.0, 2.0, 4.0, 10.0, and 20.0 ml of the 0.1 ppm standard solution to one half of a filter paper.

## V. Procedures:

### A. Sample Preparation:

#### Mercury

##### Aqueous Samples

Transfer 25 ml of liquid sample to a 250 ml Erlenmeyer flask. Add 2.5 ml of concentrated  $\text{H}_2\text{SO}_4$  and 1.3 ml of concentrated  $\text{HNO}_3$ , mixing after each addition. Add 7.5 ml of potassium permanganate solution, stir

and add additional solution if the permanganate color does not persist. Prepare a blank solution using the same amount of reagent. Add 4.0 ml of potassium persulfate solution after about 15 minutes and heat on a steam bath for two hours. Remove from the steam bath and allow the solution to cool.

#### Filters

Transfer the filter sample to a 250 ml beaker, add 50 ml of 0.1 M iodine monochloride solution and allow to stand for at least two hours with occasional stirring.

#### Silver, Lead, Chromium, Nickel and Cadmium

##### Aqueous Samples

Transfer 100 ml of sample to a 150 ml beaker. Add 3.0 ml of concentrated  $\text{HNO}_3$  and 5.0 ml of  $\text{H}_2\text{O}_2$  (30%). Digest on the hot plate until the volume is reduced to about 40 ml. Cool, transfer to a 50 ml volumetric flask and dilute to volume with water.

#### Filters

Transfer the composited filter sample to a 500 ml beaker, add 70 ml of concentrated  $\text{HNO}_3$  and digest on the hot plate for about two hours. Add 5 ml of perchloric acid (60%) and evaporate to fumes. Fume for about 5 minutes, cool and dilute with water to 50 ml in a volumetric flask.

#### B. Calibration:

##### Mercury

Standardize the mercury system by carrying aliquots of the working standard solution containing 0.05, 0.10, 0.15 and 0.20  $\mu\text{g}$  of mercury through the procedure. This is accomplished by adding 0.50, 1.0, 1.5, and 2.0 ml of the working standard solution directly to the reaction flask, diluting to 125 ml with water and proceeding with the reduction, aeration and atomic absorption measurement. (Note: The range of standardization may be extended to 0.30  $\mu\text{g}$  of mercury if necessary). Prepare a calibration curve by plotting peak height versus  $\mu\text{g}$  of mercury.

#### Silver, Lead, Chromium, Nickel, Cadmium

The working calibration standards in Section 4.a. are introduced manually into the flame or by the automatic sample injector into the graphite

furnace. By proper programming the calibration data are stored in the mini-computer system of the atomic absorption spectrometer.

C. Analysis:

Mercury

Attach a collector tube to the outlet tube of the mercury reduction apparatus (reaction flash) and adjust the stop cock to direct the carrier gas flow through the reduction apparatus. Attach the tube from the nitrogen gas supply to the inlet tube.

Add hydroxylamine hydrochloride solution with a medicine dropper to the sample solution until the pink color disappears and then add 1 ml excess. Transfer the solution (which contains 25 ml of the original liquid sample) to the reaction vessel, washing out the Erlenmeyer flask with deionized water and adding the washings to the solution. Dilute the solution to 100-125 ml with deionized water, add 10 ml of stannous chloride solution, and attach to the scrubbing system. Pass nitrogen gas through the system at about 700 ml per minute for two minutes. Adjust the stop-cock to the by-pass position, attach the collector tube to the absorption cell and flush with nitrogen gas for one minute.

Plug the Variac controller into the 110 volt line, adjust the Variac setting to 19-20, and attach the leads from the Variac controller to the resistance wiring on the collector tube. Turn on the current, and thermally release the amalgamated mercury until the recorded peak on the strip recorder returns to the base line (usually 15-20 seconds). Turn off the current, remove the collector tube from the absorption cell, and prepare for the next run.

Measure the peak height response obtained from the sample and determine the amount of mercury present by referring to the calibration curve. Correct this result by subtracting the amount of Hg found in a reagent blank carried through the whole procedure.

For the reagent blank, transfer 10.0 ml of the iodine monochloride solution used to prepare filter samples to the reaction vessel. Dilute to about 100 ml with water, and 5 ml of sodium hydroxide solution (20%), reduce with hydroxylamine hydrochloride and proceed as described for water solutions.

Silver, Lead Chromium, Nickel and Cadmium

Aspirate the sample solutions contained in the 50 ml volumetric flasks into the flame of the Perkin-Elmer Model 5000 atomic absorption spectrometer. Use the automatic sample injector to introduce samples into the graphite furnace if this mode of excitation is being used. The

instrument will automatically compare the response obtained from samples with the stored calibration data and will provide a measurement of concentration on a print-out tape.

## VI. Calculations:

### Mercury

$$\text{Concentration in Water (ppb)} = \frac{(A-B)}{V \times 1000}$$

where

A =  $\mu\text{g}$  found in sample aliquot

B =  $\mu\text{g}$  found in blank sample

V = volume of sample analyzed (ml) - 25 ml for Hg

$$\text{Filters } (\mu\text{g}/\text{m}^2) = \frac{(A - B) \times V_1}{V_2 \times F \times S}$$

where

A =  $\mu\text{g}$  found in the aliquot portion of the sample solution analyzed

B =  $\mu\text{g}$  found in the blank sample aliquot

$V_1$  = volume of sample solution (ml) - normally 50 ml

$V_2$  = volume of aliquot analyzed (ml) - normally 25 ml

F = fraction of one whole filter used as sample (normally 0.5)

S = area swiped ( $\text{m}^2$ ) - normally 1

### Other Metals

$$\text{Concentration in water } (\mu\text{g}/\ell) = \frac{(C_A - C_B)}{2} \times 1000$$

$$\text{Amount on filter } (\mu\text{g}/\text{m}^2) = \frac{(C_A - C_B) \times 50}{F \times A}$$

where

$C_A$  = Concentration ( $\mu\text{g}/\text{ml}$ ) in digested sample

$C_B$  = Concentration ( $\mu\text{g}/\text{ml}$ ) in blank

F = Fraction of filter analyzed (normally 0.5)

A = area swiped ( $\text{m}^2$ ) - normally 1.

#### VII. References:

Long, Scott, and Thompson, "Atomic Absorption Determination of Elemental Mercury Collected from Ambient Air on Silver Wool", Anal. Chem. 45, 2227 (1973).

D. L. Chase, D. L. Sgontz, E. R. Blosser, and W. M. Henry, "Summary Report on Development and Evaluation of an Analytical Method for the Determination of Total Atmospheric Mercury", NTIS No. PB-210822, 59 (1972).

Methods for Chemical Analysis of Water and Wastes, Environmental Monitoring and Support Laboratory, Office of Research and Development, EPA Cincinnati, Ohio, March 1979 Edition, Method 245.1.

Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer, September 1976.

Analytical Methods Using the HGA Graphite Furnace, Perkin-Elmer, March 1977.

# Spike Recovery Data

## A. Water

Concentration (ug/liter)											
Ag		Pb		Cr		Ni		Cd		Hg	
Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found
0	ND	0	ND	0	ND	0	ND	0	ND	0	ND
0.5	0.3	5	7	5	5	5	10	0.5	0.7	0.2	ND
1.0	0.8	10	10	10	10	10	8	1.0	0.9	0.4	0.3
2.0	1.6	20	19	20	18	20	(lost)	2.0	1.9	0.8	0.8
5.0	4.2	50	45	50	47	50	55	5.0	4.7	2.0	2.0
10	8.5	100	94	100	92	100	103	10	9.4	4.0	3.4

11

## B. Filters

Concentration (ug/liter)											
Ag		Pb		Cr		Ni		Cd		Hg	
Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found
0	ND	0	ND	0	ND	0	ND	0	ND	0	ND
0.5	ND	2.5	6	2.5	ND	2.5	ND	25	25	0.2	0.19
1.0	1.1	5	8	5	6	5	8	50	50	0.4	0.33
2.0	2.0	10	12	10	12	10	14	100	100	0.8	0.79
5.0	5.2	25	27	25	27	25	27	250	246	2.0	2.1
10	10.2	50	53	50	56	50	55	500	520	4.0	4.2

MERCURY ON FILTERS

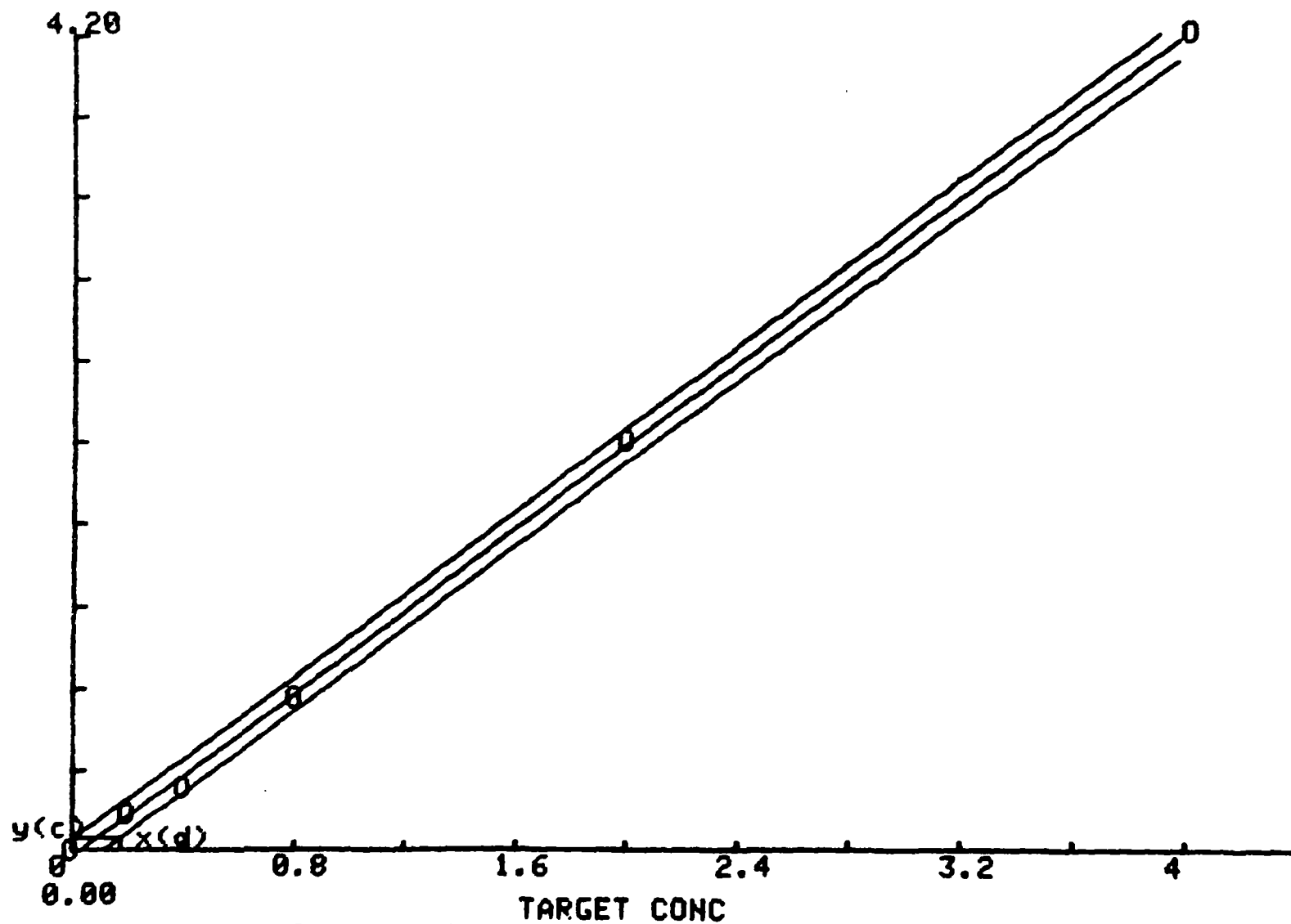
1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
0.20	0.19			
0.40	0.33			
0.80	0.79			
2.00	2.10			
4.00	4.20			

# MERCURY ON FILTERS

1  
FOUND CONC



MERCURY ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 1.2333333333 SD= 1.53057723316

FOUND CONC

MEAN= 1.2683333333 SD= 1.62278053558

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.0390210586227

SLOPE= 1.06001707456

R= 0.99978892124

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00138949914636

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.0514634962259

x(d)= 0.169620741708

# CADMIUM ON FILTERS

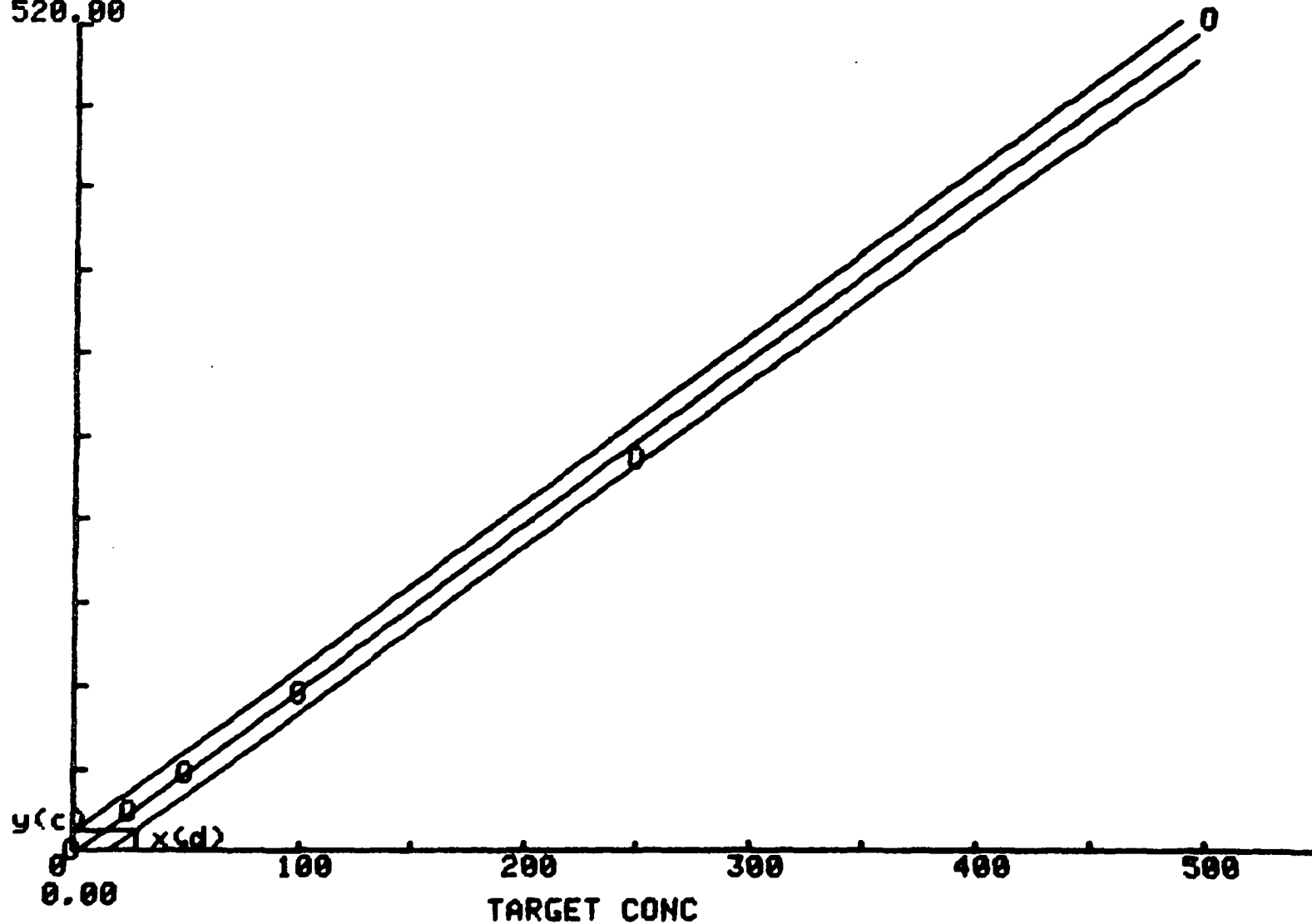
## 1 COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
25.00	25.00			
50.00	50.00			
100.00	100.00			
250.00	246.00			
500.00	520.00			

# CADMIUM ON FILTERS

1  
FOUND CONC

520.00



VERTICAL AXIS TIC INTERVAL= 52

CADMIUM ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 154.166666667 SD= 191.322154145

FOUND CONC

MEAN= 156.833333333 SD= 198.222518062

N0. RUNS 1 TOTAL X-Y ALL RUNS 6 N0. CONCENTR 6  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -2.83665338645

SLOPE= 1.03569721116

R= 0.999643347375

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 35.0278884472

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 11.5298900739

x(d)= 27.5150511892

# NICKEL ON FILTERS

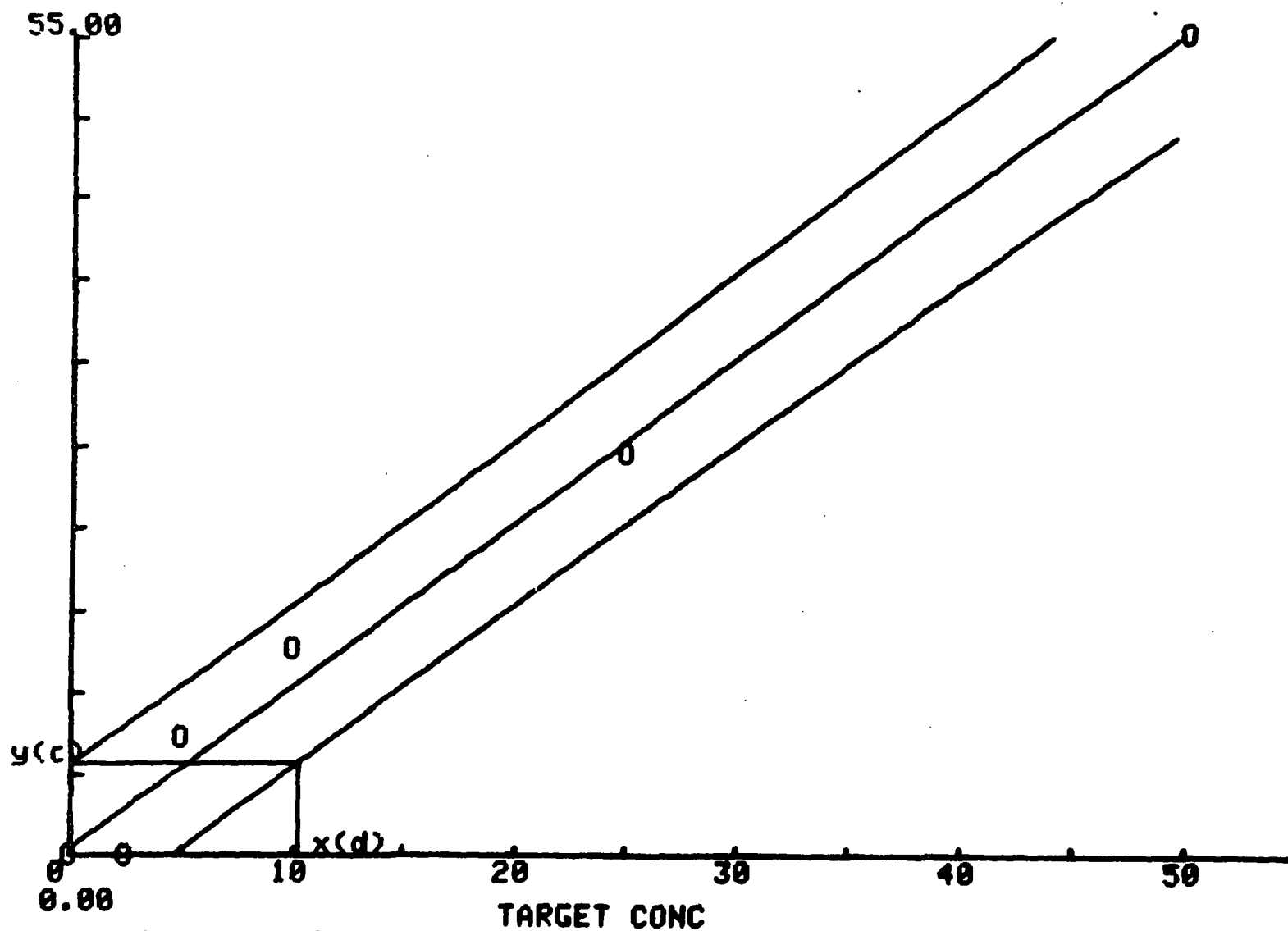
1

## COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
2.50	0.00			
5.00	8.00			
10.00	14.00			
25.00	27.00			
50.00	55.00			

# NICKEL ON FILTERS

1  
FOUND CONC



NICKEL ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 15.4166666667 SD= 19.1322154145

FOUND CONC

MEAN= 17.3333333333 SD= 21.0301371053

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.472396129767

SLOPE= 1.0936824132

R= 0.994980081191

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 5.53642572568

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 6.18402593202

x(d)= 10.207739712

TOTAL CHROMIUM ON FILTERS

1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
2.50	0.00			
5.00	6.00			
10.00	12.00			
25.00	27.00			
50.00	56.00			



TOTAL CHROMIUM ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 15.4166666667 SD= 19.1322154145

FOUND CONC

MEAN= 16.8333333333 SD= 21.6556382189

NØ. RUNS 1 TOTAL X-Y ALL RUNS 6 NØ. CONCENTR 6  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.585657370518

SLOPE= 1.12988047809

R= 0.998221178287

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 2.08366533866

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 2.9183038876

x(d)= 6.10213883257

# LEAD ON FILTERS

## 1 COMPILATION OF TARGET CONC. VS FOUND CONC.

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
2.50	6.00			
5.00	8.00			
10.00	12.00			
25.00	27.00			
50.00	53.00			

1  
FOUND CONC



LEAD ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 15.4166666667 SD= 19.1322154145

FOUND CONC

MEAN= 17.6666666667 SD= 19.5414090246

N0. RUNS 1 TOTAL X-Y ALL RUNS 6 N0. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 1.94991462721

SLOPE= 1.01946499715

R= 0.998117582439

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 1.7953898691

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 5.20246998679

x(d)= 6.27557897609

# SILVER ON FILTERS

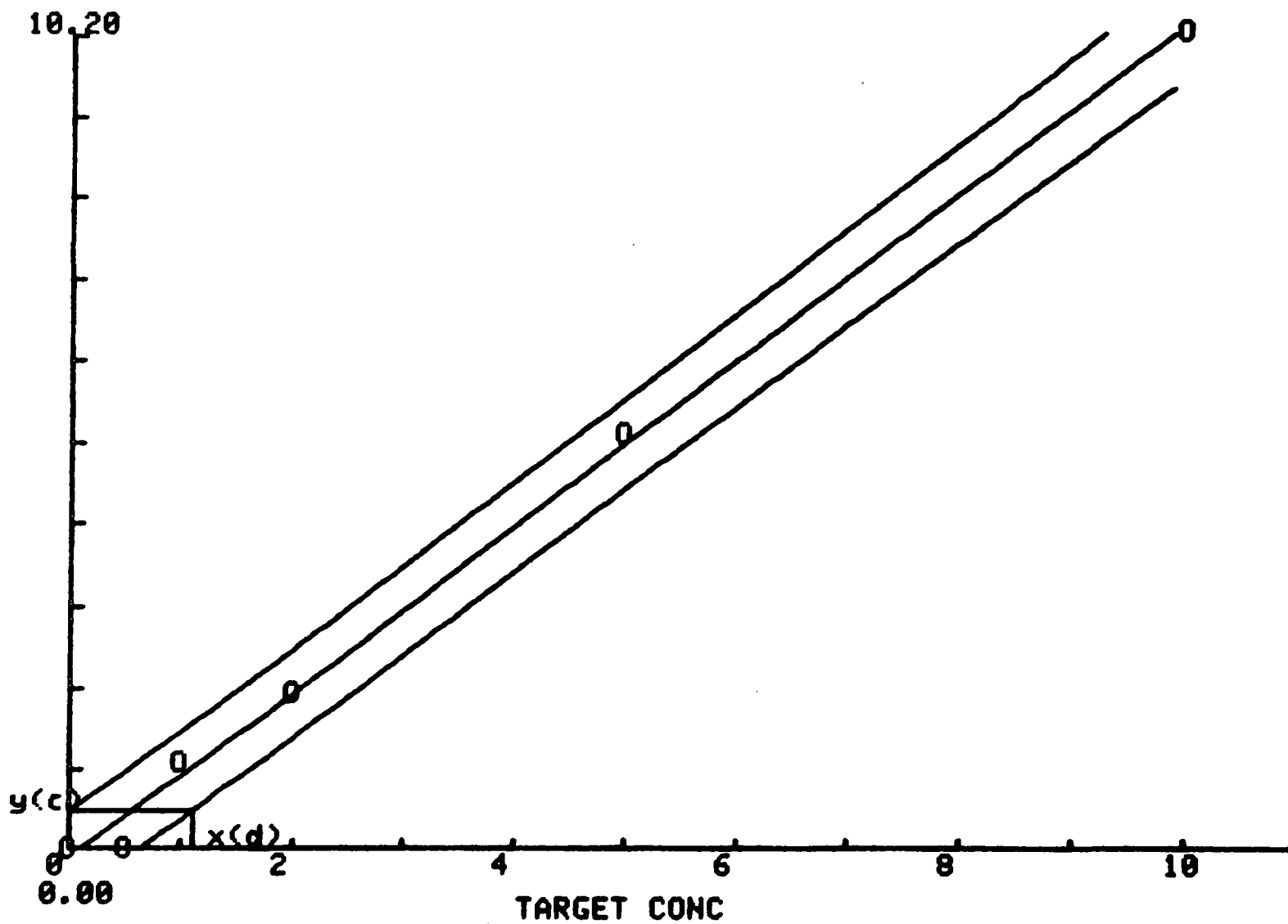
1

## COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/M2	Day 1 Found Conc. UG/M2	Day 2 Found Conc. UG/M2	Day 3 Found Conc. UG/M2	Day 4 Found Conc. UG/M2
0.00	0.00			
0.50	0.00			
1.00	1.10			
2.00	2.00			
5.00	5.20			
10.00	10.20			

# SILVER ON FILTERS

1  
FOUND CONC



VERTICAL AXIS TIC INTERVAL= 1.02

SILVER ON FILTERS

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 3.0833333333 SD= 3.8264430829

FOUND CONC

MEAN= 3.0833333333 SD= 3.9811639839

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.120034149118

SLOPE= 1.03892999431

R= 0.998553816527

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0572623790555

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.460837266725

x(d)= 1.10153175206

MERCURY IN WATER

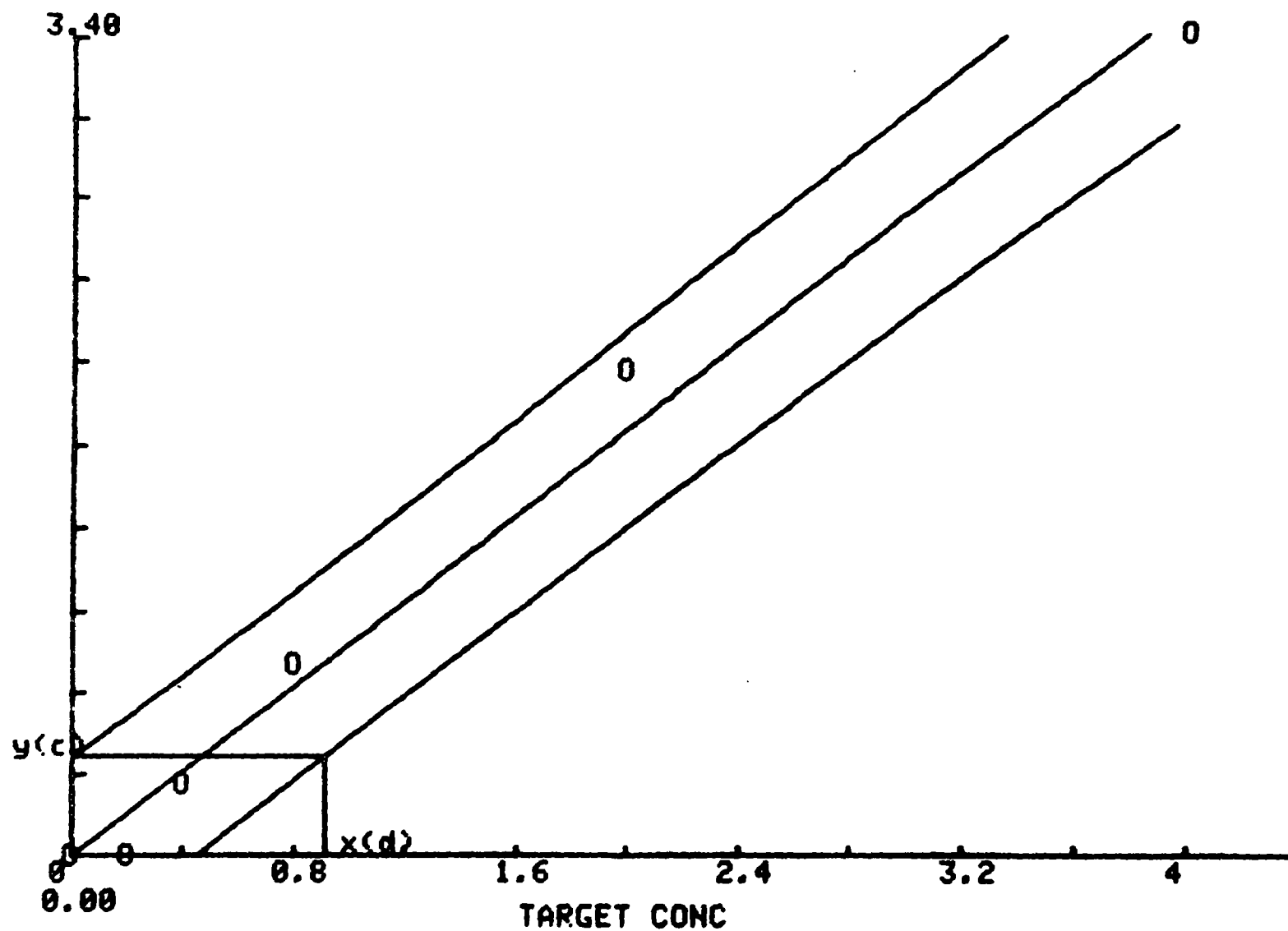
1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
0.20	0.00			
0.40	0.30			
0.80	0.80			
2.00	2.00			
4.00	3.40			

# MERCURY IN WATER

1  
FOUND CONC



VERTICAL AXIS TIC INTERVAL = 0.34

MERCURY IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 1.2333333333 SD= 1.53057723316

FOUND CONC

MEAN= 1.0833333333 SD= 1.36002450958

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.00574843483211

SLOPE= 0.883039271485

R= 0.993776064621

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0286909504838

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.405418119054

x(d)= 0.909018140513

CADMIUM IN WATER

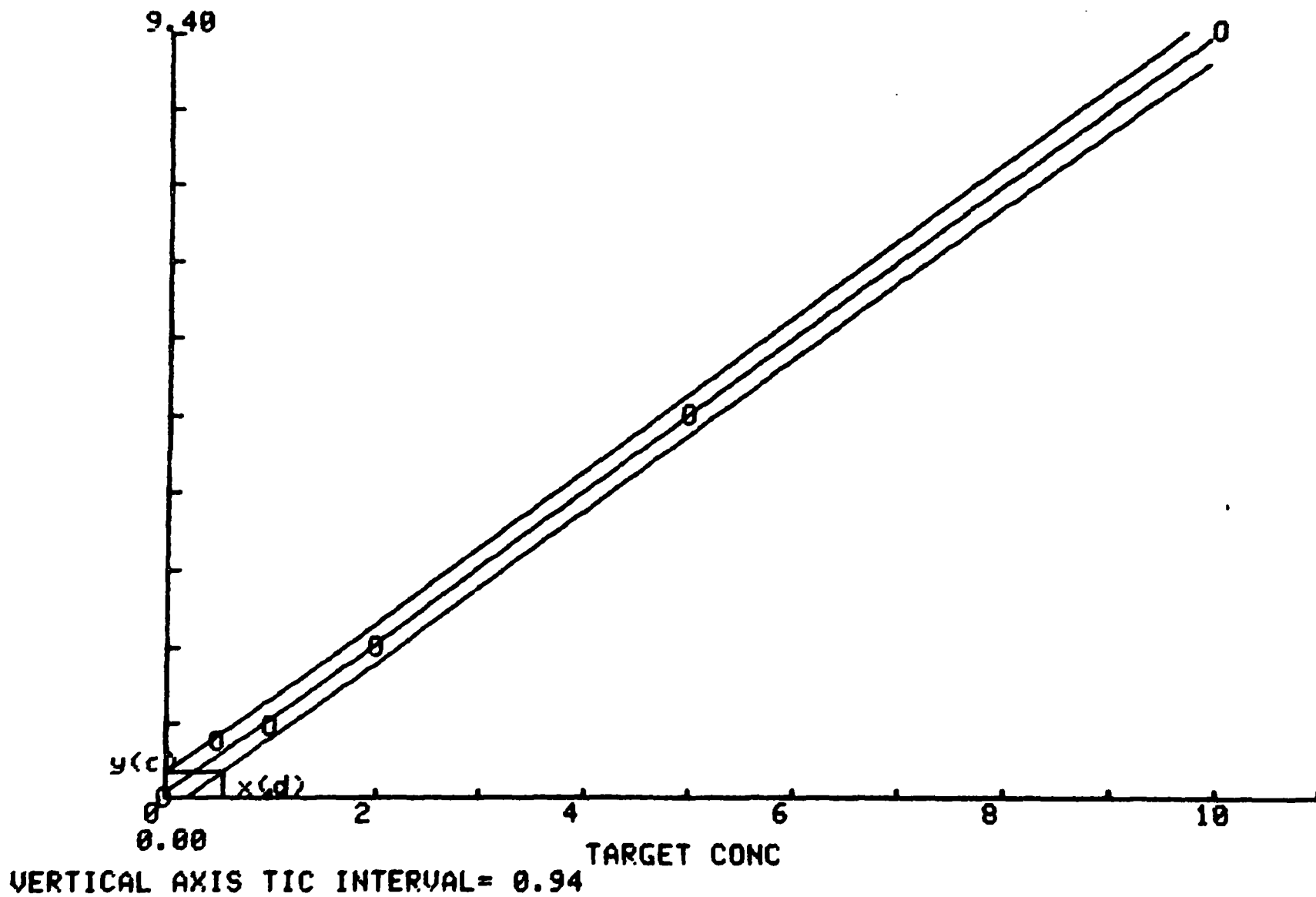
1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
0.50	0.70			
1.00	0.90			
2.00	1.90			
5.00	4.70			
10.00	9.40			

# CADMIUM IN WATER

1  
FOUND CONC



CADMIUM IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 3.0833333333 SD= 3.8264430829

FOUND CONC

MEAN= 2.9333333333 SD= 3.57024742373

N0. RUNS 1 TOTAL X-Y ALL RUNS 6 N0. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0574274331246

SLOPE= 0.932726237906

R= 0.999657289169

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0109191804212

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.311080625786

x(d)= 0.539512092829

NICKEL IN WATER

1

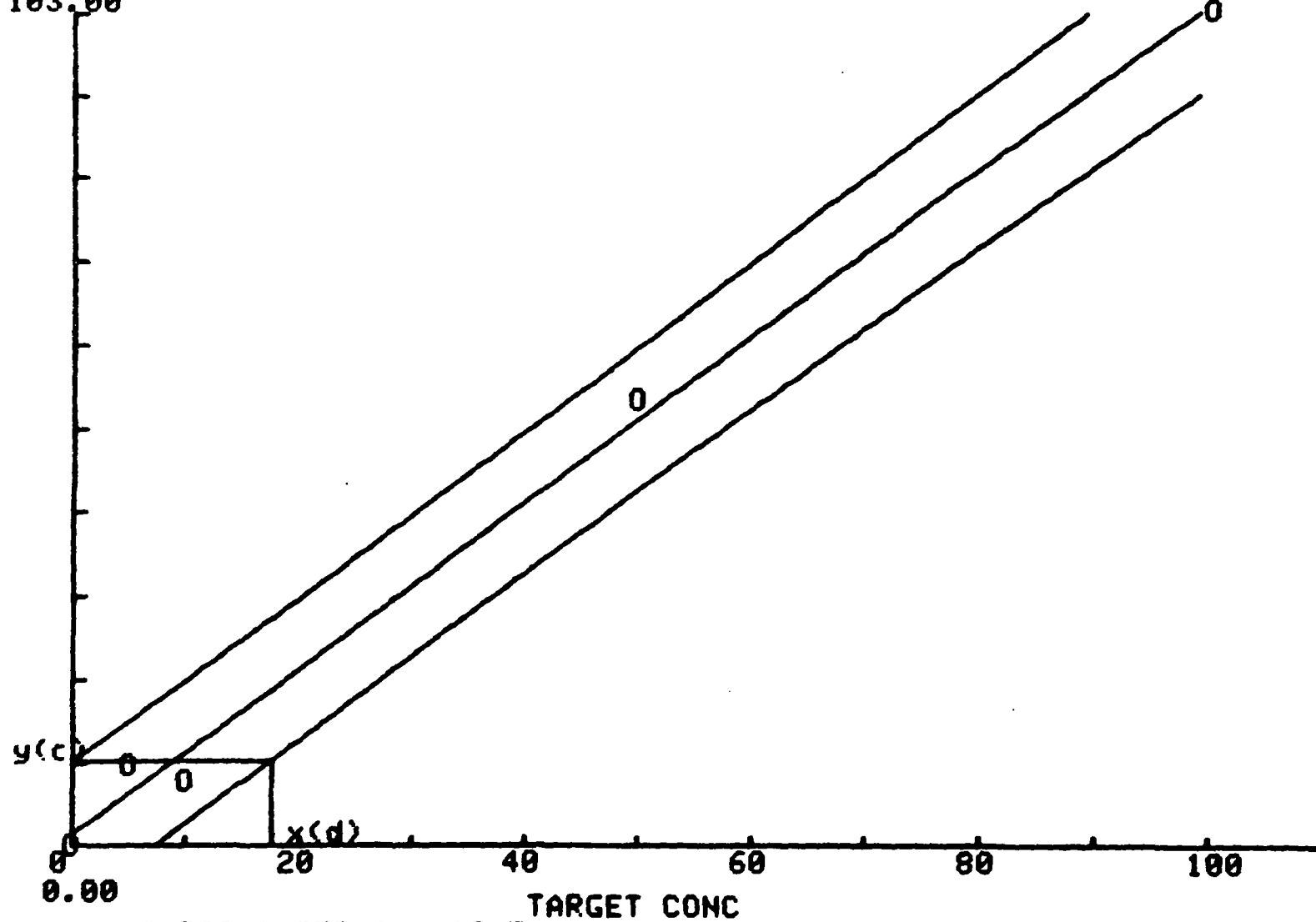
COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
5.00	10.00			
10.00	8.00			
50.00	55.00			
100.00	103.00			

# NICKEL IN WATER

1  
FOUND CONC

103.00



VERTICAL AXIS TIC INTERVAL= 10.3

NICKEL IN WATER

1

TARGET CONC

ANALYSIS OF 5 TARGET CONC-FOUND CONC POINTS

MEAN= 33 SD= 42.3674403286

FOUND CONC

MEAN= 35.2 SD= 43.5970182467

NO. RUNS 1 TOTAL X-Y ALL RUNS 5 NO. CONCENTR 5

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 1.31754874652

SLOPE= 1.02674094708

R= 0.997783508084

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 11.2219127206

COMPUTE T

D.F.= 3

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.3533525706

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 10.483045069

x(d)= 17.4550574531

TOTAL CHROMIUM IN WATER

1

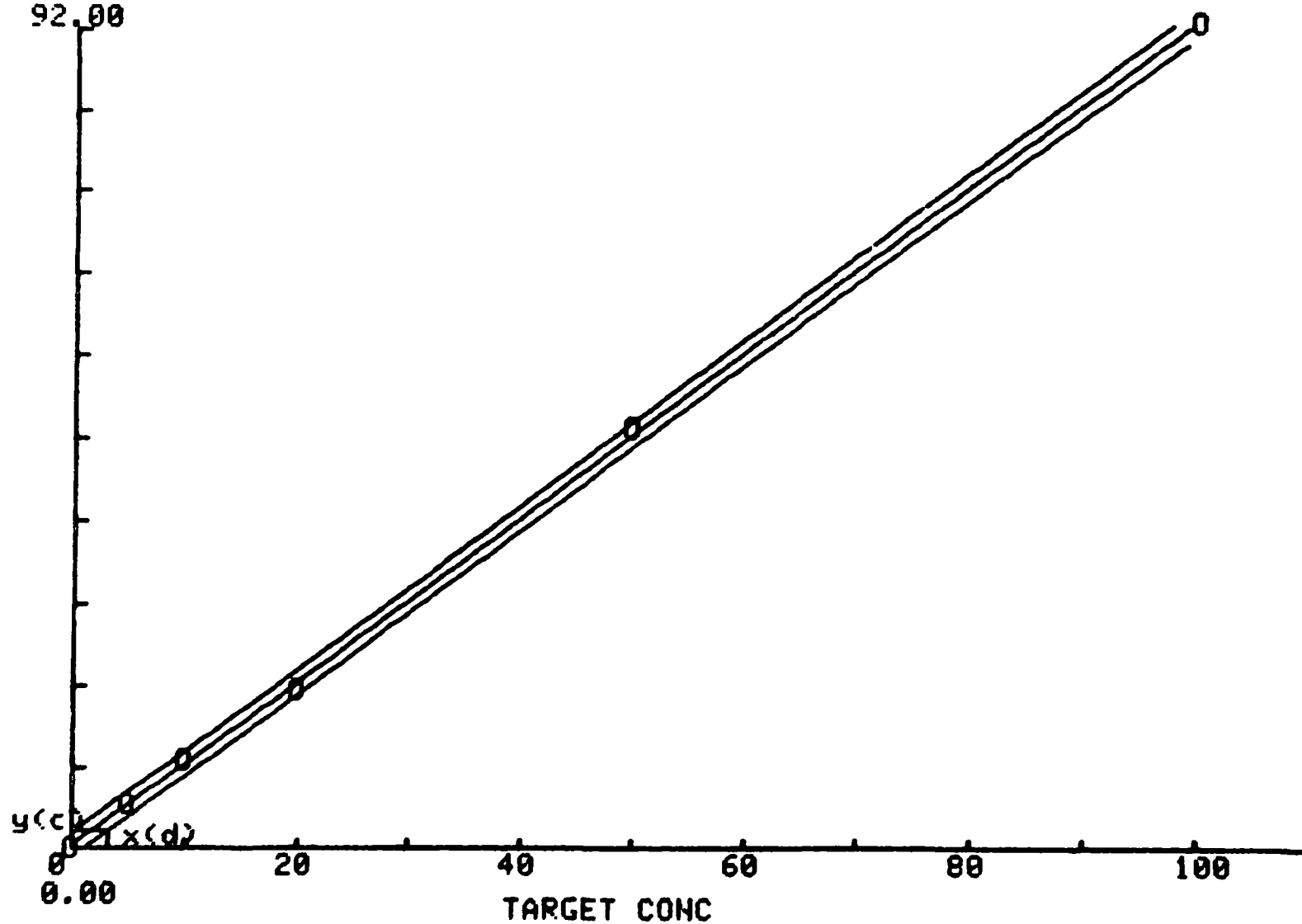
COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
5.00	5.00			
10.00	10.00			
20.00	18.00			
50.00	47.00			
100.00	92.00			

# TOTAL CHROMIUM IN WATER

1  
FOUND CONC

92.00



VERTICAL AXIS TIC INTERVAL= 9.2

TOTAL CHROMIUM IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 30.8333333333 SD= 38.264430829

FOUND CONC

MEAN= 28.6666666667 SD= 35.189013437

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.314741035857

SLOPE= 0.919521912351

R= 0.999885452143

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.354581673319

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 1.76019230441

x(d)= 3.12867970673

LEAD IN WATER

1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
5.00	7.00			
10.00	10.00			
20.00	19.00			
50.00	45.00			
100.00	94.00			

94.00



LEAD IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 30.8333333333 SD= 38.264430829

FOUND CONC

MEAN= 29.1666666667 SD= 35.4029189004

N0. RUNS 1 TOTAL X-Y ALL RUNS 6 N0. CONCENTR 6  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.656801365965

SLOPE= 0.924644280023

R= 0.999380508537

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 1.94052361982

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 4.03826544762

x(d)= 7.23740098413

SILVER IN WATER

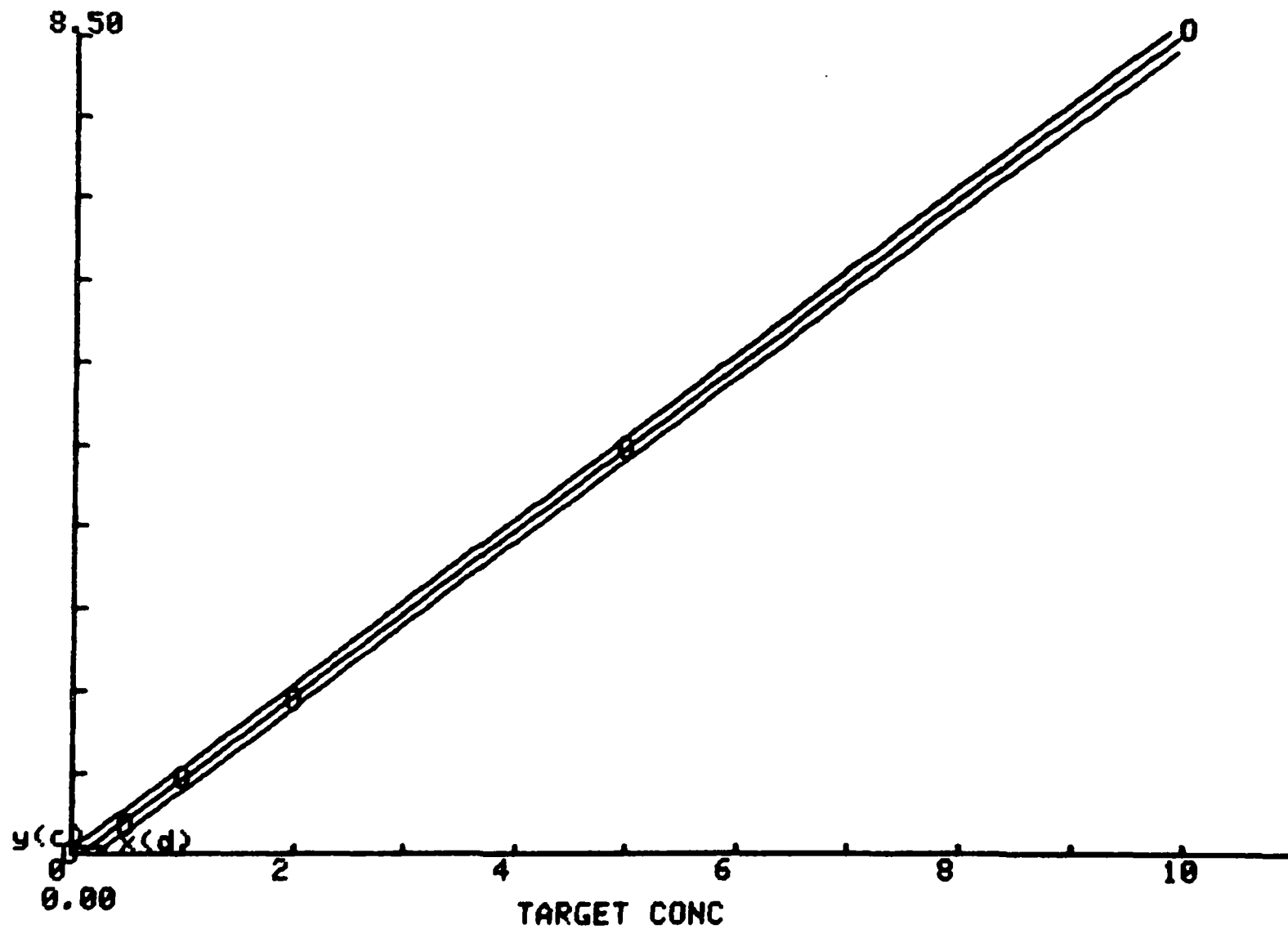
1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
0.50	0.30			
1.00	0.80			
2.00	1.60			
5.00	4.20			
10.00	8.50			

# SILVER IN WATER

1  
FOUND CONC



VERTICAL AXIS TIC INTERVAL = 0.85

SILVER IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 3.08333333333 SD= 3.8264430829

FOUND CONC

MEAN= 2.56666666667 SD= 3.27576962967

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.0726807057484

SLOPE= 0.856004553216

R= 0.999903250801

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00259533295406

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.0509828849724

x(d)= 0.287639419638

NITROGLYCERIN (NG) AND NITROCELLULOSE (NC) ON SURFACES

- I. Application: This method is applicable for the qualitative determination of nitroglycerin and nitrocellulose on surfaces.
- A. Tested Concentration Range - 0.04 to 40 micrograms per square centimeter.
  - B. Sensitivity - N/A.
  - C. Detection Limits - 0.4 micrograms per square centimeter determined by testing the above concentration range for visible color response.
  - D. Interferences - Other nitrate esters will give a positive response. The color of the surface can affect ability to distinguish the color.
  - E. Analysis Rate - Approximately 2 minutes are required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

II. Chemistry

- A. Alternate Nomenclature and Chemical Abstracts Registry Number -  
Nitroglycerin: NB; Trinitroglycerin; Glyceryl trinitrate CAS RN-55-63-0  
Nitrocellulose: NC; Cellulose nitrate Pyroxylin; colloidal cotton; soluble gun cotton. CAS RN-9004-70-0
- B. Physical and Chemical Properties - NG: mp = 13°C, decomposes at 145°C NC: decomposes on heating. Hazards: Explosive hazard, avoid heat, shock, or open flame. Toxic inhalation and skin absorption hazards exist.
- C. Chemical Reactions - Nitrite ion is cleaved in basic solution which diazotizes procaine, in acidic solution, which in turn couples with N,N-dimethyl-1-naphthylamine to produce an azo-dye. This presence or absence of color is decided by visual inspection.

III. Apparatus

- A. Instrumentation - N/A
- B. Parameters - N/A

### C. Hardware/Glassware

1. cotton-tipped swabs
2. 1 dram screw cap vials
3. volumetric flasks - 100 ml, 10 ml
4. pipettes - 50 ml, 1 ml, 10  $\mu$ l, and 5 ml graduated
5. 100-ml graduated cylinder
6. 1-cm<sup>2</sup> confined area spottest paper
7. Sprayers and reservoir bottles

### D. Chemicals

1. KOH, Analytical Reagent grade
2. ethanol, ACS grade
3. glacial acetic acid, ACS grade
4. procaine
5. N,N-dimethyl-1-naphthylamine

### E. Reagents

1. Reagent A (5% ethanolic KOH) - Weigh 5 g of KOH into the sprayer reservoir. Add 100 ml of ethanol, measured using the graduated cylinder. Swirl to dissolve KOH.
2. 50% acetic acid - water (nitrate free). Dilute to volume with deionized water.
3. Reagent B - Weigh 0.35 grams each of procaine and N,N-dimethyl-1-naphthylamine into a second sprayer reservoir. Add the 100 ml of 50% acetic acid. Cap and shake until the reagents are dissolved.

## IV. Standards

### A. Calibration Standards

1. A 4 g/l solution of NC is prepared by weighing 40 mg of NC in a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 40  $\mu$ g/cm<sup>2</sup>.
2. A 0.4 g/l solution of NC is prepared by pipetting 1 ml of the 4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.
3. A 40 mg/l solution of NC is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.

4. The NG standards having 4 g/l, 0.4 g/l, and 40 mg/l are prepared in a way analogous to Steps 1, 2, and 3. The 4 g/l solution of NG is prepared by pipetting 4 ml of a 1% NG in acetone SARM into a 10 ml volumetric flask and diluting to the mark with acetone.
5. A 100 mg/l solution of NC is prepared by pipetting 2.5 ml of the 0.4 g/l solution into a 10 ml volumetric flask, using the 5 ml graduated pipette, and diluting to the mark with acetone. This solution is used routinely for a procedure check.

B. Control Spikes - N/A.

## V. Procedure

### A. Testing the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of NC
2. Withdraw the swab and allow it to air dry.
3. Spray the swab with Reagent A and allow it to dry.
4. Spray the swab with Reagent B.
5. A positive test is indicated by a violet or red violet color and indicates that the spray solution is adequate for testing.

B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately 6 inches from the surface, apply Reagent A for approximately one second. Allow the alcohol to evaporate, then spray with Reagent B in the same manner and in the same location as Reagent A. A positive test is indicated by a violet or red violet color against a pale pink background.

VI. Calculations - N/A.

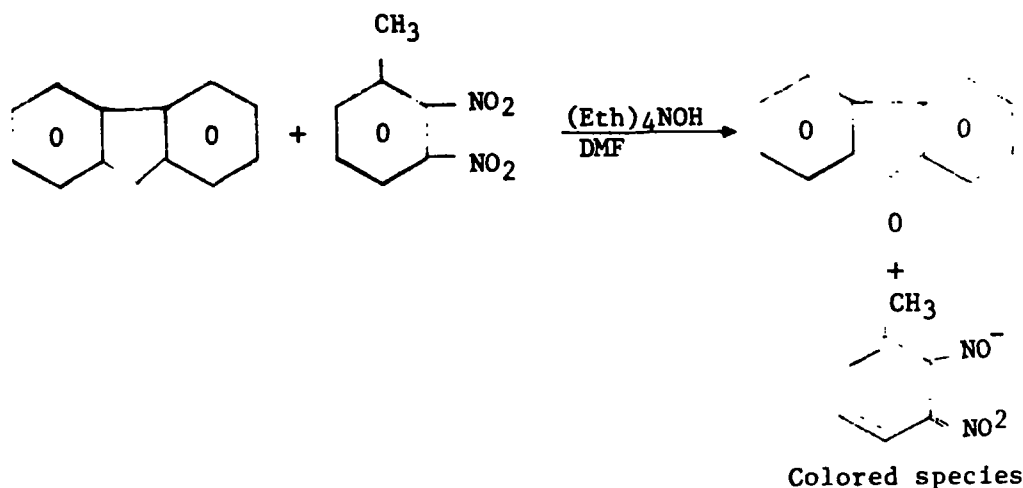
VII. References - NAVEODFAC Technical Report TR-185 Development of a Simple Portable Detection Kit for Selected Explosives, Robert E. Wyant, Battelle Columbus Laboratories, September 1977.

2,4,6-TRINITROTOLUENE (TNT), 2,4- AND 2,6-DINITROTOLUENE (DNT)  
AND 2,4,6-TRINITROPHENYLMETHYLNITRAMINE (TETRYL) ON SURFACES

- I. Application: This method is applicable for the qualitative determination of TNT, DNTs, and tetryl on surfaces.
- A. Tested Concentration Range - 0.4 to 40 micrograms per square centimeter.
- B. Sensitivity - N/A.
- C. Detection Limits - 0.4 micrograms per square centimeter determined by testing the above concentration range for visible color response.
- D. Interferences - Background color can affect ability to distinguish the color reaction.
- E. Analysis Rate - Approximately 2 minutes are required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

II. Chemistry

- A. Alternate Nomenclature and Chemical Abstracts Registry Numbers - 2,4,6-Trinitrotoluene: TNT; CAS RN 118-96-7; 2,4-Dinitrotoluene: 2,4-DNT; CAS RN 121-14-2; 2,6-Dinitrotoluene: 2,6-DNT; CAS RN 606-20-2; Tetryl: 2,4,6-Trinitrophenylmethylnitramine; 2,4,6-Tetranitro-N-methyl-N-aniline; CAS RN 479-45-8.
- B. Physical and Chemical Properties - TNT: mp = 82°C; bp = 240°C (explodes). 2,4-DNT: mp = 71°C; bp = 300°C (decomposes). 2,6-DNT: mp = 66°C; bp = 300°C (decomposed) Tetryl: mp = 131°C; bp = 187°C (explodes). Hazards: Use caution in handling. Potential explosive, toxic inhalation, and skin absorption hazards exist.
- C. Chemical Reactions - An example of the chemical reaction which occurs is given below for 2,4-DNT.



### III. Apparatus

#### A. Instrumentation - N/A.

#### B. Parameters - N/A.

#### C. Hardware/Glassware

1. cotton tipped swabs
2. 1-dram screw cap vials
3. volumetric flasks - 10 ml
4. sprayers and reservoir bottles
5. pipettes - 50 ml, 1 ml, 10  $\mu$ l, and 5 ml graduated
6. 1-cm<sup>2</sup> confined area spotting paper

#### D. Chemicals

1. DMF, ACS Grade
2. fluorene
3. 10% tetraethyl ammonium hydroxide in water.

#### E. Reagents

1. Reagent A - Weigh 0.2 g of fluorene into the sprayer reservoir. Add 10 ml of DMF, measured using the graduated cylinder. Swirl until the fluorene is dissolved.
2. Reagent B - Using a graduated cylinder, measure 100 ml of 10% tetraethylammonium hydroxide into a sprayer reservoir.

### IV. Standards

#### A. Calibration Standards

1. A 4 gram per liter solution of TNT is prepared by weighing 40 mg of TNT into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spotting filter paper, this concentration corresponds to 40  $\mu$ g/cm<sup>2</sup>.
2. A 0.4 g/l solution of TNT is prepared by pipetting 1 ml of the 4 g /l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area filter paper. This concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.
3. A 100 mg/l solution of TNT is prepared by pipetting 2.5 ml of the 4 g/l solution into a 10 ml volumetric flask using the 5 ml graduated pipette, and diluting to the mark with acetone. This solution is used routinely for a procedure check.

4. A 40 mg/l solution of TNT is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spotting filter paper, this concentration corresponds to 0.4  $\mu$ g/cm<sup>2</sup>.

5. Steps 1, 2 and 4 are repeated for 2,4-DNT, 2,6-DNT and tetryl.

B. Control Spikes - N/A.

V. Procedure

A. Validation of the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of TNT.
2. Withdraw the swab and allow it to air dry.
3. Spray the swab with Reagent A.
4. Spray the swab with Reagent B.
5. A positive test is indicated by a maroon color and indicates that the spray solution is adequate for testing.

- B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately 6 inches from the surface, apply Reagent A for approximately one second. Wait 5 seconds and apply Reagent B in the same manner and in the same location as Reagent A. A positive test is indicated by the following color reactions against a colorless background: TNT - maroon; DNTs - blue green; Tetryl - orange.

VI. Calculations - N/A.

VII. References - Colorimetric and Fluorimetric Analysis of Organic Compounds.  
M. Pesez and J. Barton, Marcel Dekker, Inc., New York, N.Y. 1974.

## TETRAZENE ON SURFACES

I. Application - This method is applicable for the qualitative determination of tetrazene on surfaces.

- A. Tested Concentration Range - 0.4 to 40  $\mu\text{g}/\text{cm}^2$ .
- B. Sensitivity - N/A.
- C. Detection Limits - 0.4  $\mu\text{g}/\text{cm}^2$  determined by testing the above concentration range for visible color response.
- D. Interferences - Background color will interfere with the ability to distinguish the color reaction.
- E. Analysis Rate - Approximately 1 minute is required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

## II. Chemistry

- A. Alternate Nomenclature and Chemical Abstracts Registry Number - Tetrazene: CAS RN
- B. Physical and Chemical Properties - mp = decomposes; bp = N/A.  
Hazard: Tetrazene is explosive, especially in the dry state. The neat solid should be stored in isopropanol and only a small portion should be dried, by placing on a clean filter paper, to prepare analytical standards.
- C. Chemical Reactions - The precise chemical complexation reaction which forms the colored species is not known.

## III. Apparatus

- A. Instrumentation - N/A.
- B. Parameters - N/A.
- C. Hardware/Glassware
  - 1. cotton tipped swabs
  - 2. 1-dram screw cap vials
  - 3. volumetric flasks - 100 ml, 10 ml
  - 4. pipettes - 1 ml, 10  $\mu\text{l}$ , and 5 ml graduated
  - 5. 10-ml and 25-ml graduated cylinders
  - 6. 1- $\text{cm}^2$  confined area spottest paper
  - 7. sprayer and reservoir bottle
  - 8. 8 oz. screw cap bottles

#### D. Chemicals

1. NaOH, Analytical Reagent grade
2. acetone, ACS grade
3. sodium nitroprusside
4. potassium ferricyanide
5. methanol, ACS grade

#### E. Reagents

1. Reagent A - Weigh 100 g of NaOH into a screw cap bottle. Add 100 ml of distilled water, cap, and swirl until dissolved.
2. Reagent B - Weigh 10 g of sodium nitroprusside into a screw cap bottle. Add 10 ml of distilled water, cap, and swirl until dissolved.
3. Reagent C - Weigh 10 g of potassium ferricyanide into a screw cap bottle. Add 10 ml of distilled water, cap, and swirl until dissolved.
4. Fresh each day, mix 10 ml each of reagents A, B, and C in a sprayer reservoir bottle. Add 30 ml of distilled water, swirl, and allow to stand for 20 minutes. Add 60 ml of acetone and swirl.

### IV. Standards

#### A. Calibration Standards

1. A 4 g/l solution of tetrazene is prepared by weighing 40 mg of tetrazene into a 10 ml volumetric flask and diluting to the mark with methanol. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 40  $\mu$ g/cm<sup>2</sup>.
2. A 0.4 g/l solution of tetrazene is prepared by pipetting 1 ml of the 4 g/l solution into a 10 ml volumetric flask and diluting to the mark with methanol. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.
3. A 100 mg/l solution of tetrazene is prepared by pipetting 2.5 ml of the 0.4 g/l solution into a 10 ml volumetric flask, using a 5 ml graduated pipette, and diluting to the mark with methanol. This solution is used routinely for a procedure check.
4. A 40 mg/l solution of tetrazene is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask and diluting to the mark with methanol. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 0.4  $\mu$ g/cm<sup>2</sup>.

B. Control Spikes - N/A.

V. Procedure

A. Testing the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of tetrazene.
2. Withdraw the swab and allow it to air dry.
3. Spray the swab with the color reagent.
4. A positive test is indicated by a red-orange color and indicates that the spray is adequate for testing.

B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately six inches from the surface, apply the spray reagent for about one second. A positive test is indicated by a red-orange color against a yellow background.

VI. Calculations - N/A.

VII. References - personal communication with R. E. Wyant, Battelle, Columbus Laboratories.

2,4,6-TRINITRORESORCINOL (STYPHNIC ACID) AND  
LEAD STYPHNATE ON SURFACES

- I. Application: This method is applicable for the qualitative determination of styphnic acid and lead styphnate on surfaces.
- A. Tested Concentration Range - 0.4 to 40  $\mu\text{g}/\text{cm}^2$ .
  - B. Sensitivity - N/A.
  - C. Detection Limits - 0.4  $\mu\text{g}/\text{cm}^2$  determined by testing the above concentration range for visible color response.
  - D. Interferences - Background color will interfere with ability to see the color reaction.
  - E. Analysis Rate - Approximately 1 minute is required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

II. Chemistry

- A. Alternate Nomenclature and Chemical Abstracts Registry Number - 2,4,6-Trinitroresorcinol: TNR; Styphnic Acid; CAS RN-82-71-3
- B. Physical and Chemical Properties - mp = 175°C;  
Hazards: TNR is explosive and toxic. Only small quantities of the neat material should be handled with the analyst wearing gloves. Avoid heating the material.
- C. Chemical Reactions - The precise chemical complexation reaction of the Styphnate ion which forms the colored species is not known.

III. Apparatus

- A. Instrumentation - N/A.
- B. Parameters - N/A.
- C. Hardware/Glassware
  - 1. cotton tipped swabs
  - 2. 1 dram screw cap vials
  - 3. volumetric flasks - 100 ml, 10 ml
  - 4. pipettes - 1 ml, 10  $\mu\text{l}$ , and 5 ml graduated
  - 5. 100 ml graduated cylinder
  - 6. 1  $\text{cm}^2$  confined area spottest paper
  - 7. sprayers and reservoir bottles

8. filter paper - 11 cm Whatman 42
9. glass funnel
10. 125 ml Erlenmeyer flask

D. Chemicals

1. NaOH, Analytical Reagent Grade
2. Stannous chloride trihydrate
3. Acetone, ACS Grade

E. Reagents

The reagent for this test must be prepared fresh at least every 3 days.

Weigh 5 g of NaOH and 10 g of stannous chloride trihydrate into an Erlenmeyer flask. Add 100 ml of distilled water, measured using the graduated cylinder, and swirl until all NaOH is dissolved. Filter the reagent through a Whatman 42 paper into the sprayer reservoir.

IV. Standards

A. Calibration Standards

1. A 4 g/l solution of TNR is prepared by weighing 40 mg of TNR into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 40  $\mu$ g/cm<sup>2</sup>.
2. A 0.4 g/l solution of TNR is prepared by pipetting 1 ml of the 4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.
3. A 100 mg/l solution of TNR is prepared by pipetting 2.5 ml of the 0.4 g/l solution into a 10 ml volumetric flask, using a 5 ml graduated pipette, and diluting to the mark with acetone. This solution is used routinely for a procedure check.
4. A 40 mg/l solution of TNR is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask, and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 0.4  $\mu$ g/cm<sup>2</sup>.

B. Control Spikes - N/A.

## V. Procedure

### A. Testing the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of TNR.
2. Withdraw the swab and allow it to air dry.
3. Spray the swab with the color reagent.
4. A positive test indicated by a yellow-brown color and indicates that the spray solution is adequate for testing.

- B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately six inches from the surface, apply the spray reagent for about one second. A positive test is indicated by a yellow-brown color.

### VI. Calculations - N/A.

- VII. References - personal communication with R. E. Wyant, Battelle, Columbus Laboratories.

CYCLOTETRAMETHYLENETETRANITRAMINE (HMX) AND  
PENTAERYTHRITE TETRANITRATE (PETN) ON SURFACES

I. Application: This method is applicable for the qualitative determination of HMX and PETN on surfaces.

A. Test Concentration Range - 0.4 to 40 micrograms per square centimeter.

B. Sensitivity - N/A.

C. Detection Limits - 0.4 micrograms per square centimeter determined by testing the above concentration range for visible color response.

D. Interferences - Other nitrate esters will give a positive response. The color of the surface can affect ability to see the color.

E. Analysis Rate - Approximately 2 minutes are required to conduct this analysis. Limitations on the number of analyses that can be performed by this method are generally dictated by the proximity of the surfaces to be analyzed.

II. Chemistry

A. Alternate Nomenclature and Chemical Abstracts Registry Number -  
Cyclotetramethylenetetranitramine: HMX; homocyclonite; 1,3,5,7-tetranitro-1,3,5,7-tetrazacyclooctane; CAS RN-479-45-8

Pentaerythrite Tetranitrate: PETN; penta; pentrit; nitro pentaerythrite CAS RN-78-11-5

B. Physical and Chemical Properties - HMX: mp = 276-7°C; bp = 337°(explodes)  
PETN: mp = 141.3°C; bp = 210°C (explodes). Hazards: Potential explosive, toxic inhalation, and skin absorption hazards exist. Use caution in handling.

C. Chemical Reactions - Nitrate ion is cleaved by zinc which diazotizes procaine in acidic solution, which in turn couples with N,N-dimethyl-1-naphthylamine to produce an azo-dye. The presence or absence of color is decided by visual inspection.

III. Apparatus

A. Instrumentation - N/A.

B. Parameters - N/A.

### C. Hardware/Glassware

1. cotton-tipped swabs
2. volumetric flasks - 100 ml, 10 ml
3. sprayers and reservoir bottles
4. 100-ml graduated cylinder
5. pipettes - 50 ml, 1 ml, 10  $\mu$ l, and 5 ml graduated
6. 1 cm<sup>2</sup> confined area spottest paper
7. 1 dram screw cap vials

### D. Chemicals

1. toluene, ACS grade
2. glacial acetic acid, ACS grade
3. zinc dust
4. procaine
5. N,N-dimethyl-1-naphthylamine

### E. Reagents

1. Reagent A - Weigh 10 grams of zinc dust into the sprayer reservoir. Add 100 ml of toluene, measured in the graduated cylinder. The zinc will be a dispersion upon shaking, but will not be dissolved.
2. 50% acetic acid - 50 ml of glacial acetic acid into a 100 ml volumetric flask which is partially filled with deionized water (nitrite free). Dilute to volume with deionized water.
3. Reagent B - Weigh 0.35 grams each of procaine and N,N-dimethyl-1-naphthylamine into a second sprayer reservoir. Add the 100 ml of 50% acetic acid. Cap and shake until the reagents are dissolved.

## IV. Standards

### A. Calibration Standards

1. A 4 gram per liter solution of PETN is prepared by weighing 40 mg of PETN in a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 40  $\mu$ g/cm<sup>2</sup>.
2. A 0.4 gram per liter solution of PETN is prepared by pipetting 1 ml of the 4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 4  $\mu$ g/cm<sup>2</sup>.
3. A 100 mg/l solution of PETN is prepared by pipetting 2.5 ml of the 0.4 g/l solution into a 10 ml volumetric flask, using the 5 ml graduated pipette and diluting to the mark with acetone. This solution is used routinely for a procedure check.

4. A 40 mg/l solution of PETN is prepared by pipetting 1 ml of the 0.4 g/l solution into a 10 ml volumetric flask and diluting to the mark with acetone. When 10  $\mu$ l are spotted on confined area spottest paper, this concentration corresponds to 0.4  $\mu$ g/cm<sup>2</sup>.
5. Steps 1, 2, and 4 are repeated for HMX.

B. Control Spikes - N/A.

V. Procedure

A. Testing the Spray Solution

1. Dip a cotton swab into a vial containing a 100 mg/l (0.01%) solution of PETN.
2. Withdraw the swab and allow it to air dry.
3. Spray the swab with Reagent A, shaking well before using.
4. Spray the swab with Reagent B.
5. A positive test is indicated by a violet or red violet color and indicates that the spray solution is adequate for testing.

B. Surface Testing - The surface to be tested is not prepared in any way. Holding the sprayer approximately 6 inches from the surface, apply Reagent A, after shaking, for approximately one second. Wait 15 seconds and apply Reagent B in the same manner and in the same location as Reagent A. A positive test is indicated by a violet or red violet color against a pale pink background.

VI. Calculations - N/A.

VII. References - NAVEODFAC Technical Report TR-185 "Development of a Simple Portable Detection Kit for Selected Explosives", Robert E. Wyant, Battelle Columbus Laboratories, September 1977.

DETERMINATION OF 2,4,6-TRINITRORESORCINOL  
(STYPHNIC ACID) IN WATER

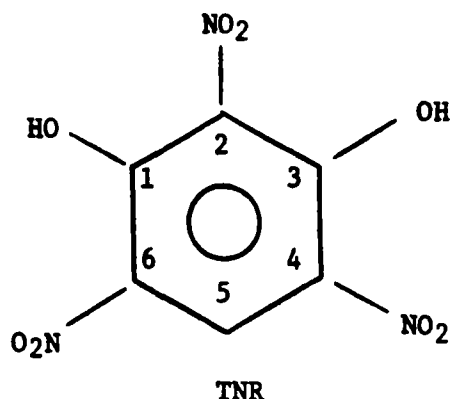
I. APPLICATION

This method is used to quantitatively determine the concentration of styphnic acid (TNR) in aqueous samples.

- A. Tested Concentration Range. The concentration range is from 20 to 500  $\mu\text{g}/\ell$ .
- B. Sensitivity Limits. The signal to noise ratio was 10:1 for 2.5 ng of TNR.  
At detection limit 1 ng =  $8.5 \times 10^{-5}$  AU
- C. Detection Limits. The detection limit has been determined to be 32  $\mu\text{g}/\text{liter}$ .
- D. Interferences. Compounds which adsorb UV light at 254nm and coelute with TNR will interfere.
- E. Analysis Rate. Approximately fifteen samples can be analyzed by one worker in an eight hour day.

II. SEPARATION SYSTEM AND CHEMISTRY

TNR, whose chemical structure is shown below, has a high UV absorbance at 254nm, so it can be detected using a UV detector (with a fixed wavelength of 254nm) with high sensitivity. The separation is performed using a reversed-phase high performance liquid chromatographic system with a  $\text{C}_{18}$ -bonded-phase column. In order to avoid ionization of the TNR, some acetic acid is added to the mobile phase, thereby protonating the TNR.



CAS RN # -- 82-71-3

Melting Point -- 175°C

Boiling Point -- N/A

Handling Hazards -- TNR is explosive and toxic. Only small quantities of the neat material should be handled with the analyst wearing gloves. Avoid heating the material.

### III. APPARATUS

A. Instrumentation. Waters Model 45 HPLC pump, a Rheodyne Model 7120 injection valve, an LDC UV III Monitor (1203) detector and a Linear Model 235 strip-chart recorder.

#### B. HPLC Parameters

1. Column: 25 x 0.46 cm ID SS
2. Column Packing: Spherisorb ODS
3. Particle Diameter: 5  $\mu$ m
4. Mobile Phase: 15% Acetonitrile/85% Water + 0.5% Acetic Acid
5. Flow Rate: 1.5 ml/min.
6. Inlet Pressure: ~2000 psi
7. Temperature: ambient
8. Injection Volume: 50  $\mu$ l
9. Detector Wavelength: 254 nm
10. Detector Sensitivity: 0.04 AUFS
11. Recorder Span: 10 mV FS
12. Chart Speed: 0.5 cm/min
13. Retention Time for TNR: 3.50 min

C. Hardware/Glassware

1. Millipore all glass filter apparatus
2. Nuclepore polyester filters, 0.2  $\mu$ m pore size, 47 mm diameter
3. 1000-ml graduated cylinder
4. 250-ml graduated cylinder
5. 10-ml pipet
6. 1-ml pipet
7. 50-ml volumetric flask
8. 10-ml volumetric flask
9. 10- $\mu$ l Hamilton syringe
10. 25- $\mu$ l Hamilton syringe
11. 100- $\mu$ l syringe - for injector
12. Syringe filtration apparatus
13. Millipore filters, 0.45  $\mu$ m, type GS, 13 mm diameter
14. 2-oz bottle with Teflon lined screw cap
15. Labels
16. 2-dram screw cap vials with Teflon cap liners
17. Refrigerator

D. Chemicals.

1. Acetonitrile, DIG grade, Burdick and Jackson
2. HPLC water, Millipore Model RO4, organic free, deionized
3. Glacial acetic acid, Baker reagent grade
4. Methanol, DIG, Burdick and Jackson

E. Reagents. None

VI. STANDARDS

A. Calibration standards.

1. Stock. (1000 mg/l) weigh 50 mg of styphnic acid into a 50 ml volumetric flask and dilute to the mark with methanol.
2. Diluted Stock. (100 mg/l) Pipet 1 ml of stock into a 10 ml volumetric flask and dilute to the mark with 0.5% of acetic acid in water.

3. Working. Prepare calibration standards containing 0, 0.05, 0.1, 0.2, 0.5, and 1 mg/l in 0.5% acetic acid as described below:

<u>Concentration (mg/l)</u>	<u>Dilution Procedure</u>
0	0 ml of diluted stock in 100 ml
0.05	0.05 ml of diluted stock in 100 ml
0.1	0.1 ml of diluted stock in 100 ml
0.2	0.2 ml of diluted stock in 100 ml
0.5	0.5 ml of diluted stock in 100 ml
1	1 ml of diluted stock in 100 ml

- B. Control Spikes: Control spiked are equivalent to working standards since the method involves direct injection.

## V. PROCEDURE

- A. Sample Handling/Preparation. Filter the sample through a 0.45  $\mu$ m Millipore filter prior to analysis and store refrigerated in a Teflon lined screw capped vial until analyzed.
- B. Calibration. Inject 50  $\mu$ l of each TNR calibration standard 4 times into the HPLC system. Record the retention time, concentration and peak height. Also inject a mobile phase blank (50  $\mu$ l).
- C. Analysis. Inject 50  $\mu$ l of sample into the HPLC system. Record the retention time and peak height for peak eluting at the retention time for TNR.

## VI. CALCULATIONS

Use the calibration data for TNR to establish a linear regression curve (concentration vs. peak height). Interpolate sample concentration from these curves.

The relationship between found component concentration and sample volume of water is as follows:

$$\mu\text{g}/\ell \text{ water} = \text{ng}/50\mu\text{l} \times 20$$

VII. REFERENCES

None

VIII. RESPONSE DATA

$\mu\text{g}/\ell$ TNR	Peak Height (mm)			
	Day 1	Day 2	Day 3	Day 4
0	1.3	0.6	0	0.5
20	16	11	12	12
50	29	22	27	24
100	43	45	40	43
200	71	83	83	85
500	192	196	210	200

TNR IN WATER

RUNS 1 2 3 4

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	3.39	2.74	-4.79	0.66
20.00	12.97	17.20	22.23	18.96
50.00	54.47	49.01	56.01	50.35
100.00	99.17	101.06	96.55	100.03

TNR IN WATER

RUNS 1 2 3 4

TARGET CONC

ANALYSIS OF 16 TARGET CONC-PEAK AREA POINTS

MEAN= 42.5 SD= 38.9015852291

PEAK AREA

MEAN= 2.175 SD= 1.47851276626

NØ. RUNS 4 TOTAL X-Y ALL RUNS 16 NØ. CONCENTR 16

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.596696035242

SLOPE= 0.0371365638767

R= 0.977111079273

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.105991189428

COMPUTE T

D.F.= 14

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

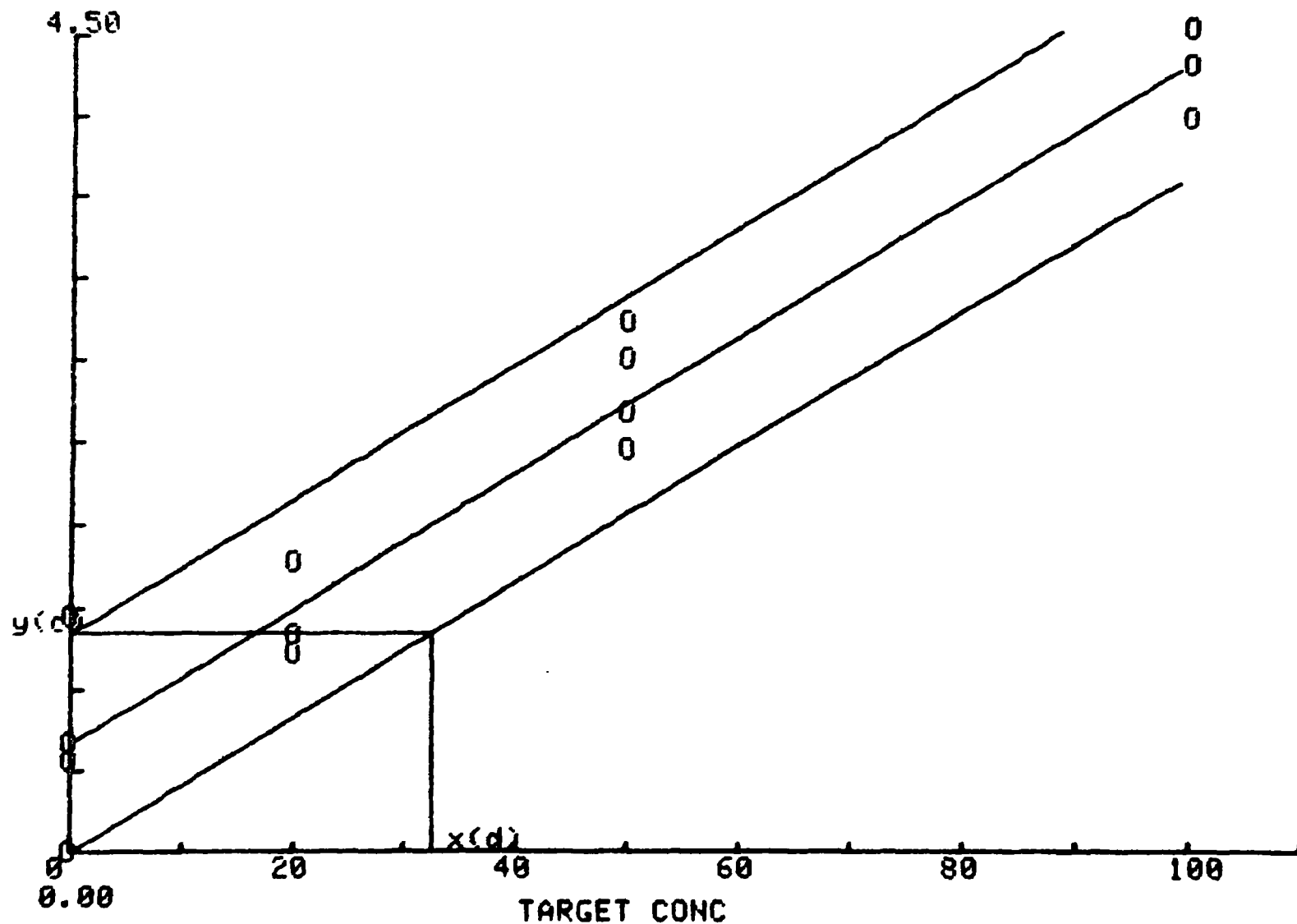
t= 1.7613101065

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 1.20949378794

x(d)= 32.4504911485

INR IN WATER  
 RUNS 1 2 3 4  
 PEAK AREA



VERTICAL AXIS TIC INTERVAL = 0.45

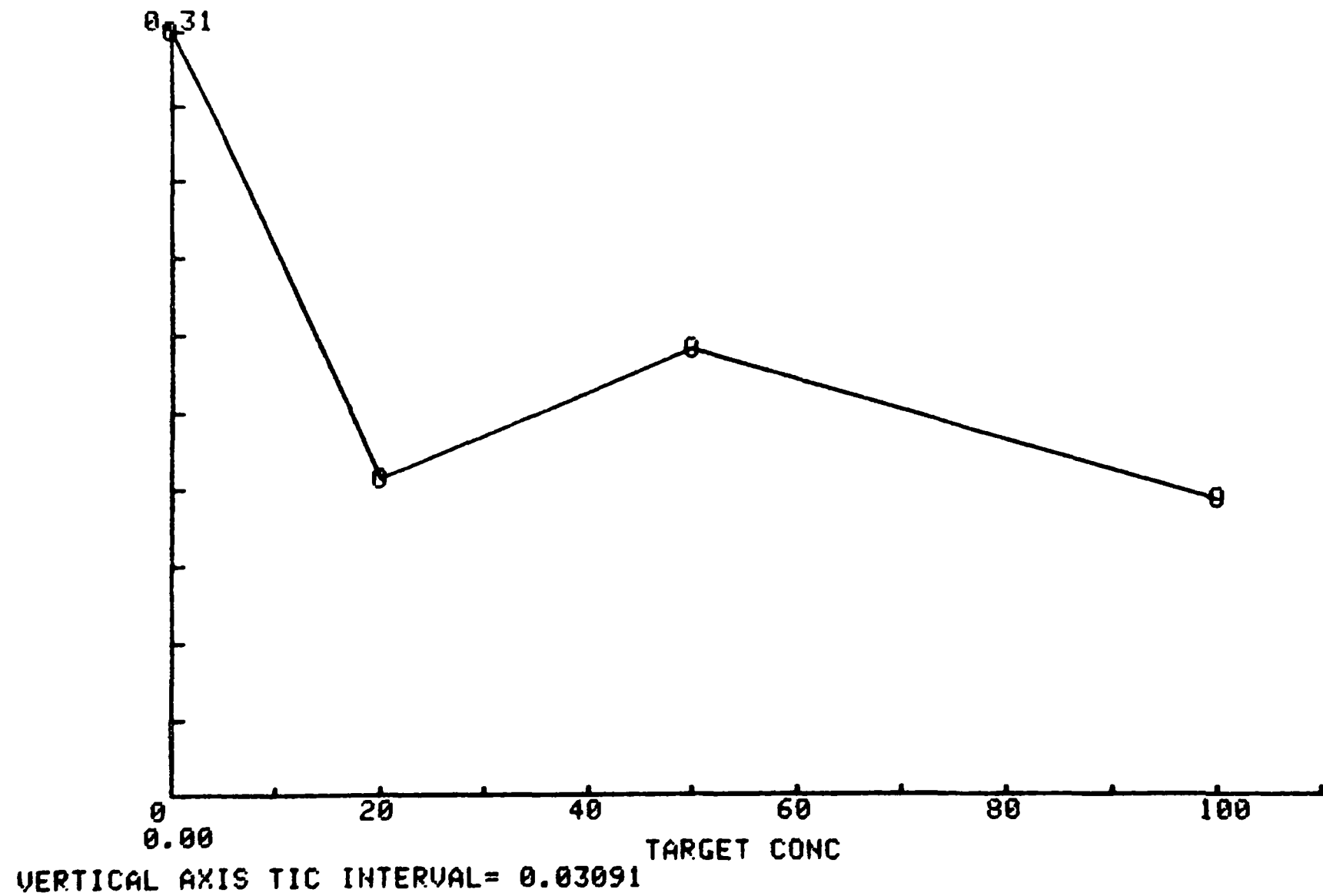
TNR IN WATER

RUNS 1 2 3 4

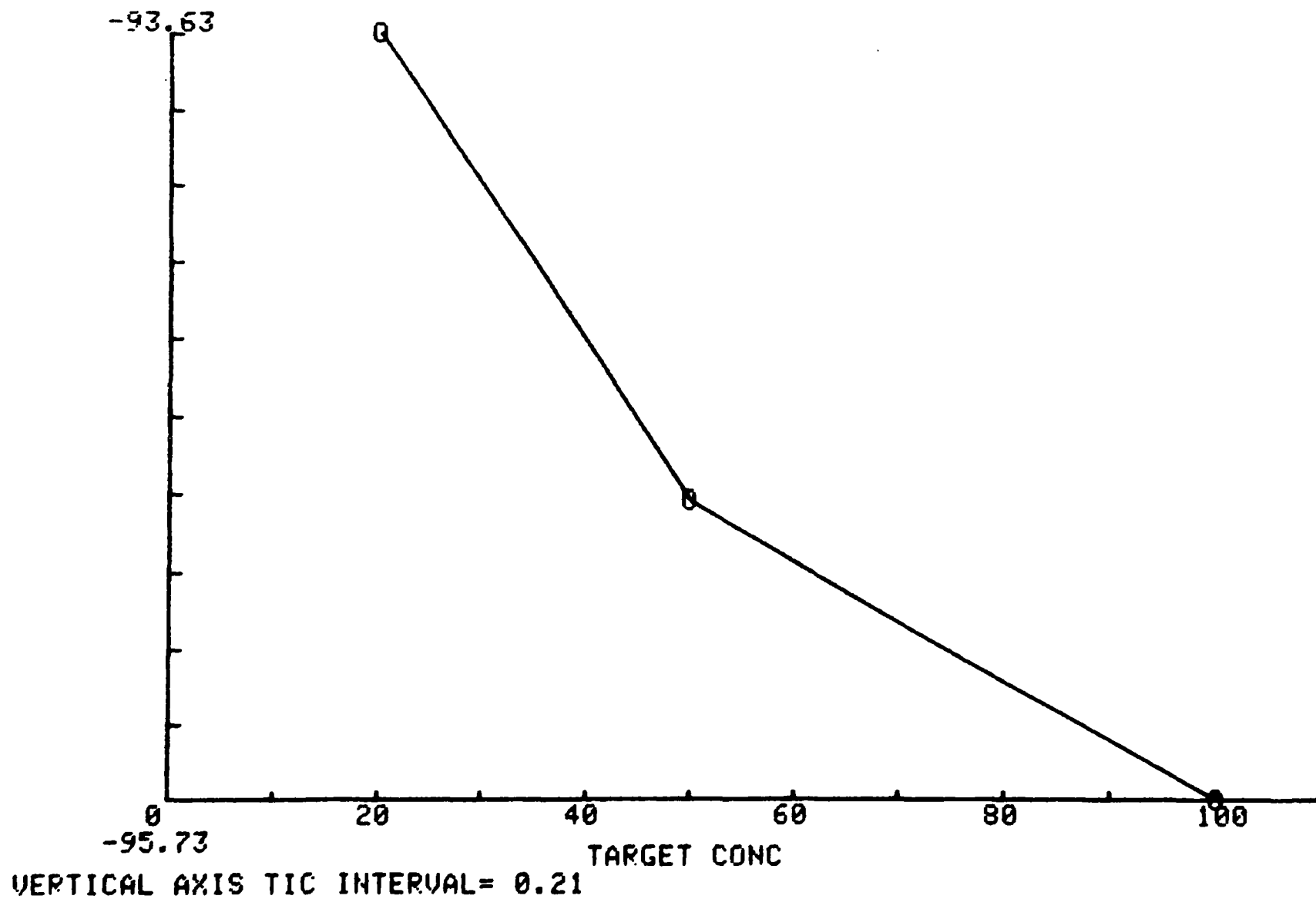
STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION

Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.00	0.50	2.14		430.30
20.00	17.84	2.23	-10.80	12.49
50.00	52.46	1.92	4.92	3.65
100.00	99.20	1.11	-0.80	1.12

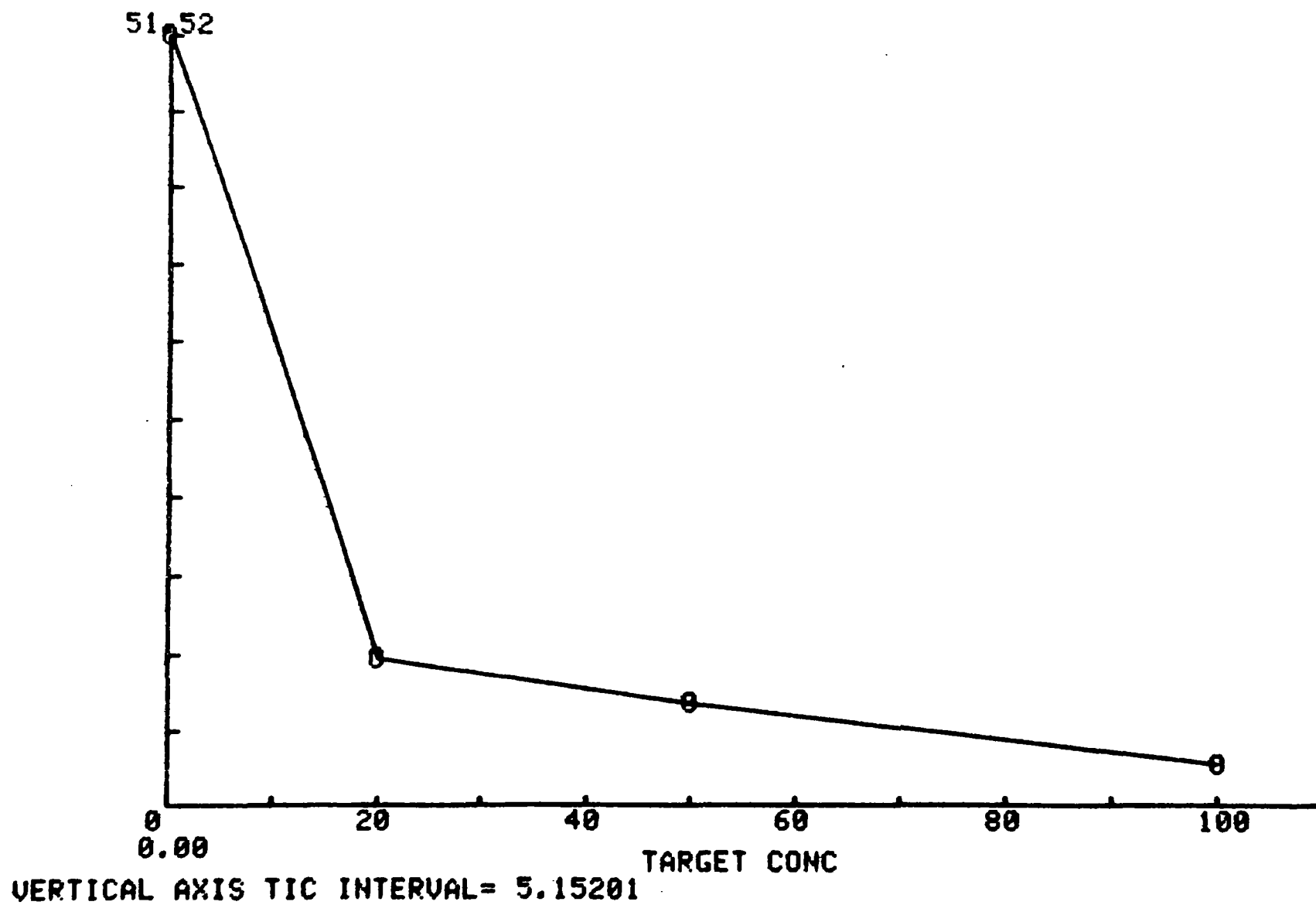
1NR IN WATER  
RUNS 1 2 3 4  
STANDARD DEVIATION



TNP IN WATER  
RUNS 1 2 3 4  
MEAN PERCENT INACCURACY



TNR IN WATER  
RUNS 1 2 3 4  
PERCENT IMPRECISION



TNR IN WATER

RUNS 1 2 3 4

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	12.30	5.53	-7.08	-0.28
20.00	20.65	18.52	21.96	17.61
50.00	56.84	47.11	58.27	48.29
100.00	95.82	93.89	101.84	101.97
200.00	173.77	205.64	193.81	204.22
500.00	510.62	499.31	501.20	498.18

TNR IN WATER  
RUNS 1 2 3 4  
TARGET CONC  
ANALYSIS OF 24 TARGET CONC-PEAK AREA POINTS

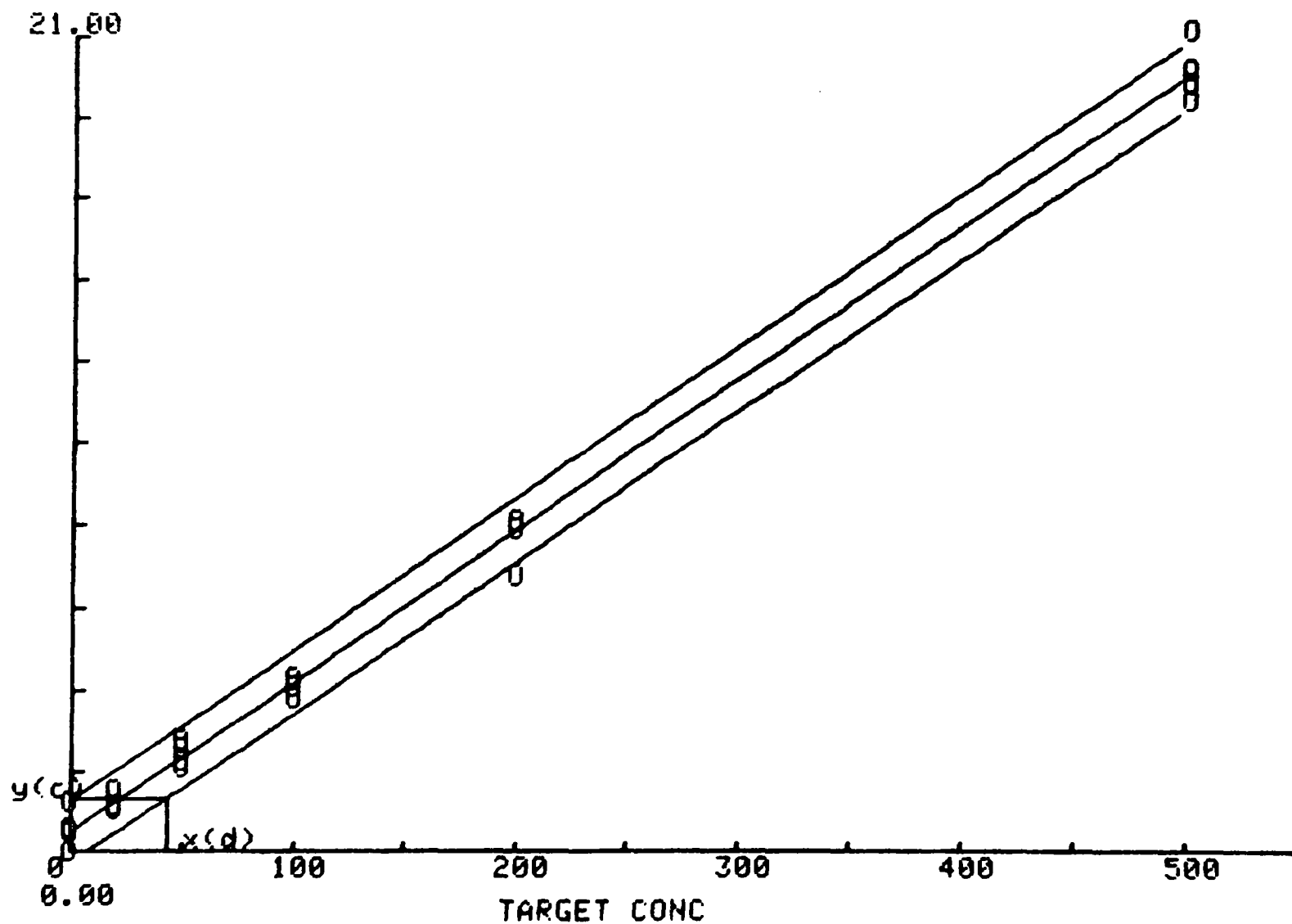
MEAN= 145 SD= 175.325783714

PEAK AREA  
MEAN= 6.125 SD= 6.80186994749

N0. RUNS 4 TOTAL X-Y ALL RUNS 24 N0. CONCENTR 24  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.51224893918  
SLOPE= 0.0387086280057  
R= 0.997758056822  
MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.216635334965  
COMPUTE T  
D.F.= 22  
ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)  
.1  
t= 1.71713909197  
REPLICATES ON UNKNOWN SAMPLE 1  
y(c)= 1.33951833223  
x(d)= 42.5942304591

1NR IN WATER  
 RUNS 1 2 3 4  
 PEAK AREA



VERTICAL AXIS TIC INTERVAL= 2.1

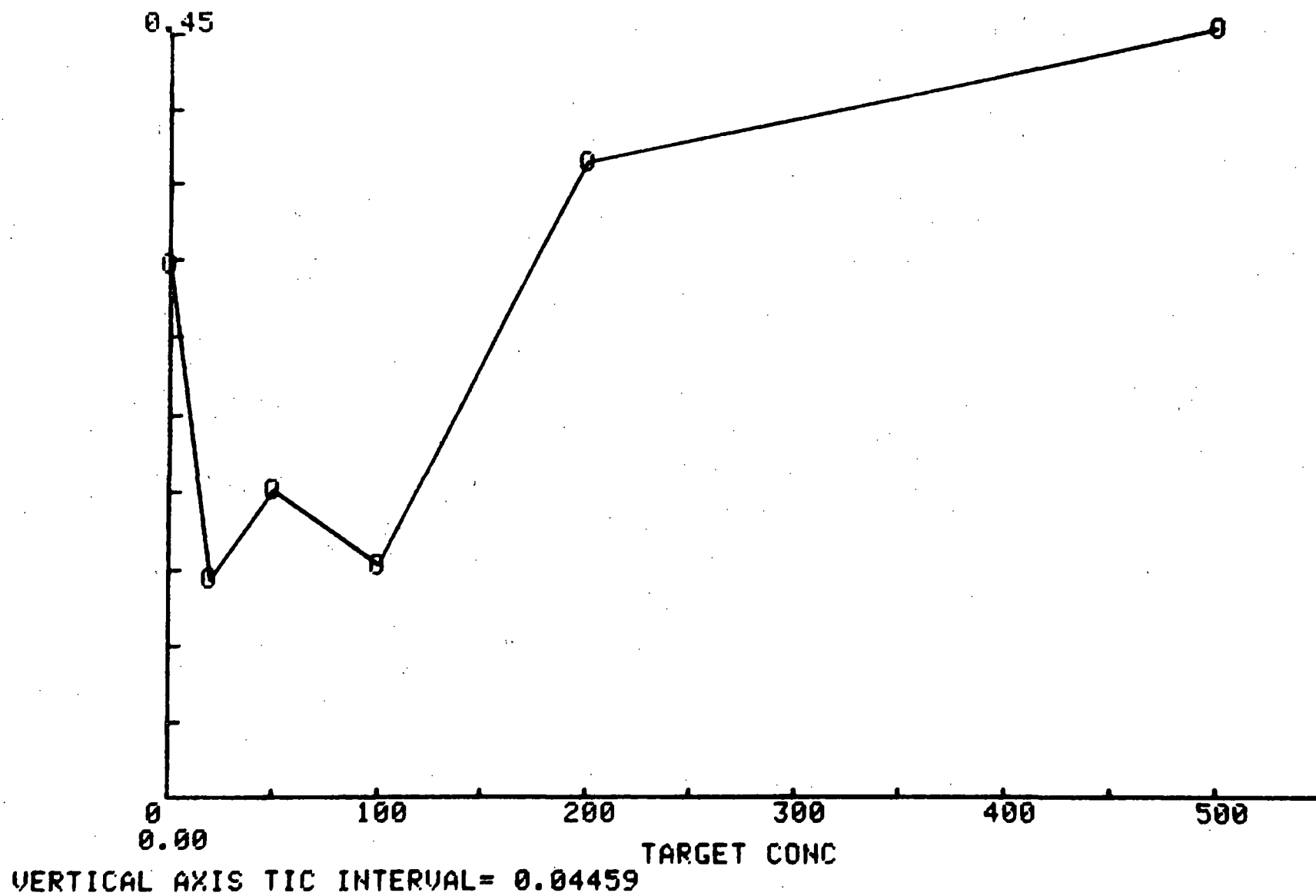
TNR IN WATER

RUNS 1 2 3 4

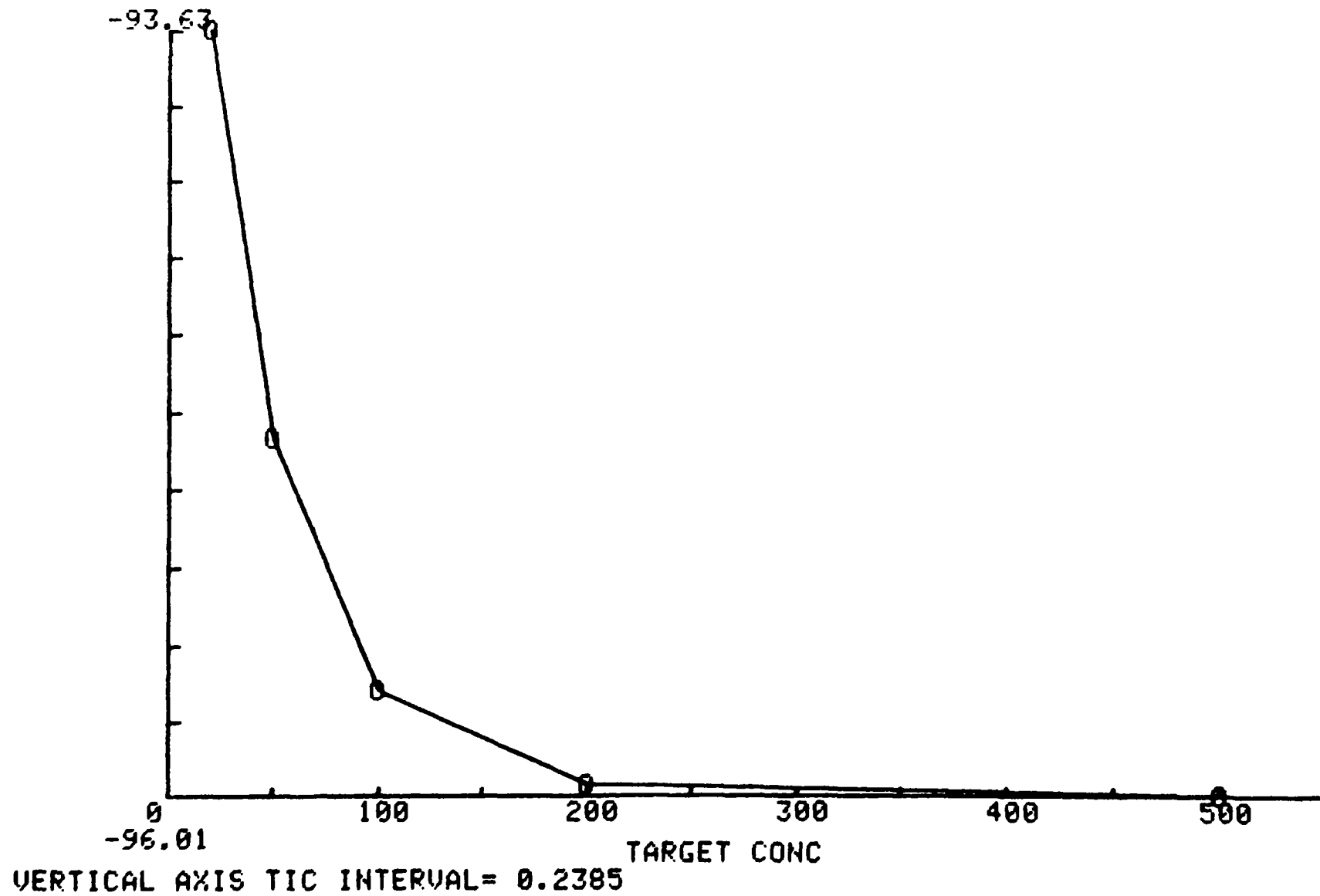
STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION

Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.00	2.62	4.77		182.26
20.00	19.69	1.14	-1.56	5.81
50.00	52.63	3.31	5.26	6.30
100.00	98.38	2.39	-1.62	2.43
200.00	194.36	8.49	-2.82	4.37
500.00	502.33	3.27	0.47	0.65

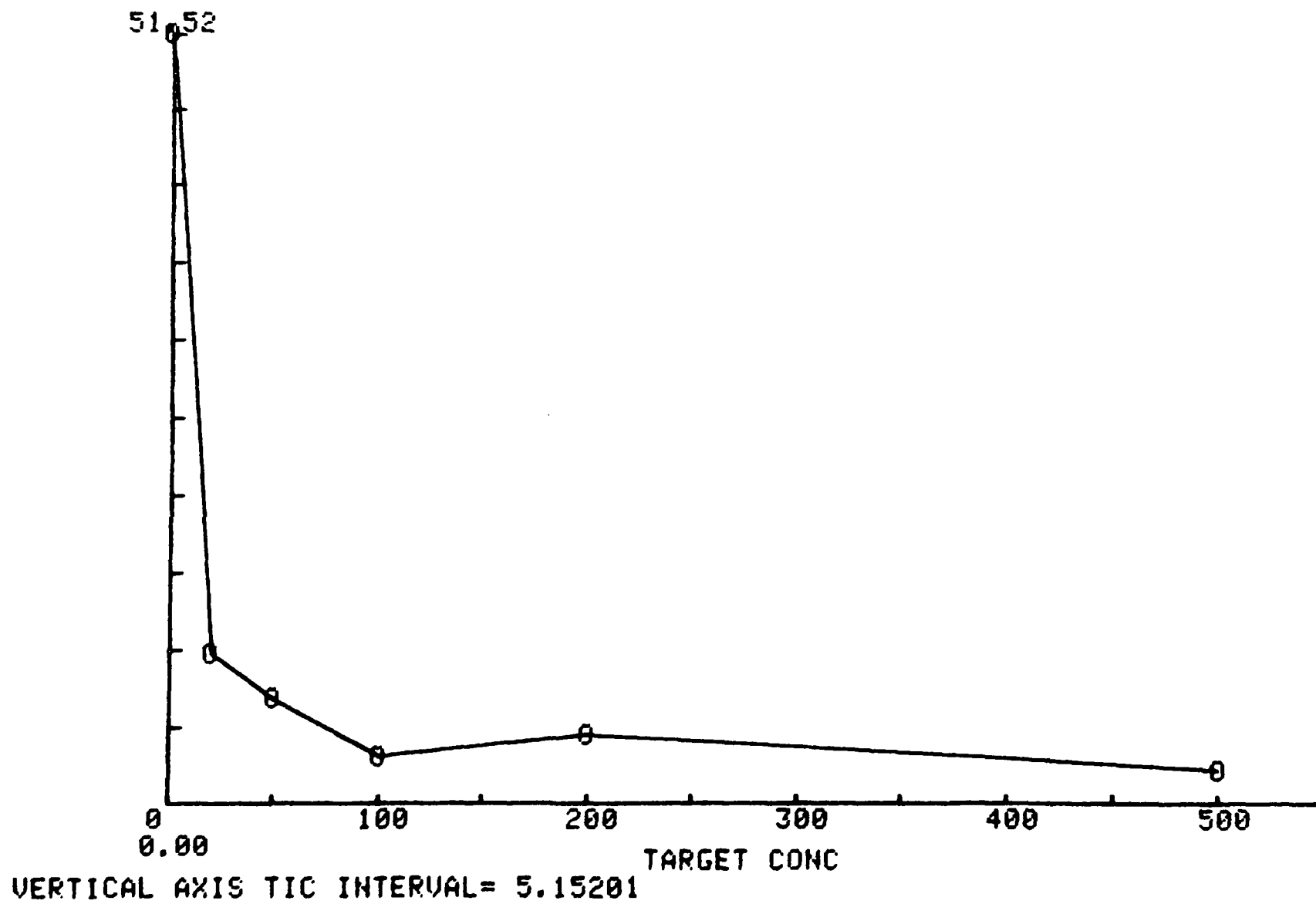
INR IN WATER  
RUNS 1 2 3 4  
STANDARD DEVIATION



INH IN WATER  
RUNS 1 2 3 4  
MEAN PERCENT INACCURACY



TNR IN WATER  
RUNS 1 2 3 4  
PERCENT IMPRECISION



TNR IN WATER

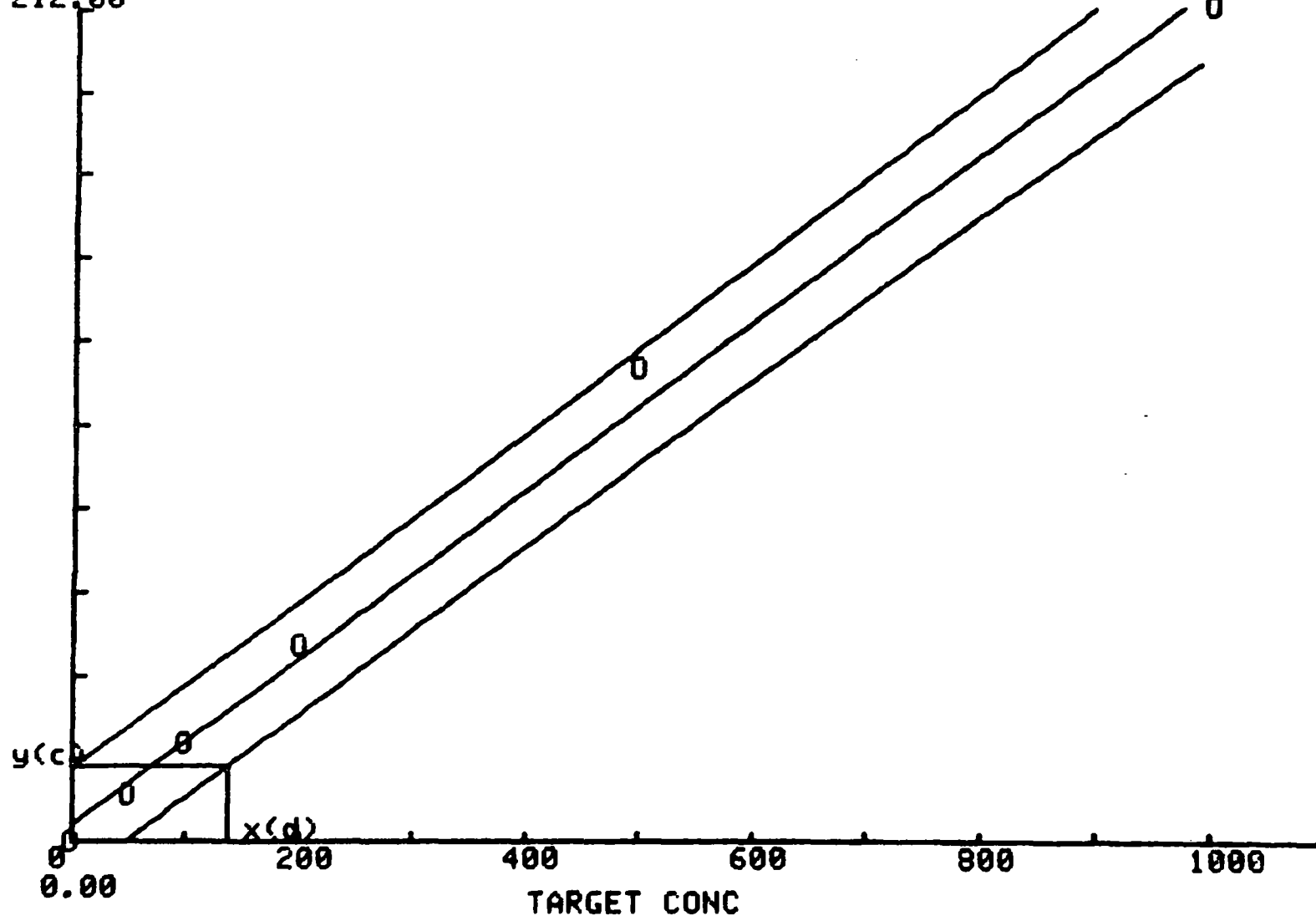
1

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	-19.58			
50.00	36.77			
100.00	97.81			
200.00	215.20			
500.00	543.90			
1000.00	975.89			

TNR IN WATER  
1  
PEAK AREA

212.00



VERTICAL AXIS TIC INTERVAL= 21.2

TNR IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-PEAK AREA POINTS

MEAN= 308.33333333 SD= 382.64430829

PEAK AREA

MEAN= 69.833333333 SD= 81.673537126

NO. RUNS 1 TOTAL X-Y ALL RUNS 6 NO. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 4.16903813318

SLOPE= 0.21296528173

R= 0.997752219201

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 37.4428002277

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 19.022561111

x(d)= 137.03470759

DETERMINATION OF HMX IN WATERI. APPLICATION

This method is used to quantitatively determine the concentration of HMX (1,3,5,7-tetranitro-1,3,5,7-tetracyclo octane) in aqueous samples.

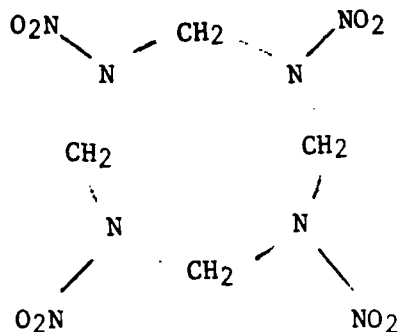
- A. Tested Concentration Range. The concentration range analyzed is 8.0 µg/l to 200 µg/l in water.
- B. Sensitivity Limits. The signal to noise ratio was 11:1 for 2 ng injected of HMX.

All the detection limit, 1 ng of HMX =  $1.4 \times 10^{-4}$  AU

- C. Detection Limits. Employing the method of Hubeaux and Vos, the detection limit for HMX in water was determined to be 8.0 µg/l (lowest level standard).
- D. Interferences. Components which elute with the same retention time as HMX and absorb UV light (254 nm) will interfere.
- E. Analysis Rate. Approximately twenty samples can be analyzed by a worker in an 8 hour day after instrument calibration.

II. CHEMISTRY

Potential explosive, toxic inhalation and skin absorption hazards exist. Chemical structure for HMX:



Alternate nomenclature - cyclotetramethylene tetranitramine; 1, 3, 5, 7- tetra-  
nitro-1, 3, 5, 7-tetracyclooctane; homocyclonite

CAS RN # - 479-45-8

Melting point - 276-7°C

Boiling point - 337°C (explodes)

### III. APPARATUS

- A. Instrumentation. An Altex Model 100A pump equipped with an Altex Model 420 microprocessor. An LDC UV III Monitor (1203) detector, a Rheodyne Model 7120 injection valve with a 100  $\mu$ l loop, and a Varian Model 9176 recorder. A Spectra Physics SP 4000 data system.
- B. HPLC Parameters.
  - 1. Column: Spherisorb 5 micron ODS, 250x4.6 mm.
  - 2. Mobile Phase: Acetonitrile/water, 40/60, v/v.
  - 3. Flow: 1 ml/min.
  - 4. Temperature: 25°C
  - 5. Detector wavelength and sensitivity: 254 nm, 0.004 AUFS
  - 6. Recorder Span: 10 mv/full scale.
  - 7. Injection volume: 100  $\mu$ l.
  - 8. Retention time for HMX:  $9.8 \pm 0.2$  minutes.
- C. Hardware/Glassware.
  - 1. Microspatula.
  - 2. 50, 100, 250 ml volumetric flasks.
  - 3. 4 ounce bottles with Teflon lined screw caps
  - 4. 2 dram vials with Teflon lined screw caps
  - 5. Pasteur pipets (5 3/4") and bulbs.
  - 6. 5 ml pipet and bulb.
  - 7. Millipore solvent filtering apparatus.
  - 8. Millipore filters, 0.45 $\mu$ , type HA, 47 mm.
  - 9. 100  $\mu$ l Hamilton syringe.
  - 10. Refrigerator and freezer.
- D. Chemicals.
  - 1. Acetone, DIG grade, Burdick and Jackson.
  - 2. Millipore, Milli-Q purified water.
- E. Reagents. None

### IV. STANDARDS

- A. Calibration Standards.
  - 1. Stock. Ten milligrams of HMX are transferred to a 50 ml volumetric flask. The flask is diluted to the mark with acetone, and its contents are mixed well.

7. Silica gel TLC plates, Whatman LK20F.
8. Oven.
9. Filter paper to line TLC tank.
10. 254 nm UV lamp.

D. Chemicals.

1. Butyl acetate, ACS grade.
2. Petroleum ether (30-60°C), Burdick and Jackson.
3. Acetone, Burdick and Jackson.
4. Potassium hydroxide, ACS grade.
5. Procaine, reagent grade.
6. N,N-dimethyl-1-naphthylamine, reagent grade.
7. Acetic acid, glacial, ACS grade.
8. Distilled water.

E. Reagents

- A. 10% KOH in water. Dissolve 10 grams of KOH in distilled water and dilute to a final volume of 100 milliliters.
- B. Modified Griess reagent. Dissolve 0.35 grams each of procaine and N,N-dimethyl-1-naphthylamine in 50% aqueous acetic acid solution.

IV. STANDARDS

Prepare individual solutions containing each compound of interest by placing 10 milligrams of the compound in a 100 milliliter volumetric flask and diluting to the mark with acetone. Mix the contents thoroughly, place in an eight ounce Teflon lined screw capped bottle, and store refrigerated.

V. PROCEDURE

- A. Sample Collection and Preparation. (1) Building surfaces. Place 1.5 milliliters of acetone in a 2 dram vial. Mark a one meter square area on the surface to be sampled. Use cotton swabs (Q-tips) wetted with acetone to swab five 20

VII. REFERENCES

None.

Spike Recovery Data for WaterHMX in water

<u>N</u>	<u>Target Value ng/ml</u>	<u>Found Value ng/ml</u>			
4	8.0	7.5	7.5	7.5	7.5
4	20	19	19	20	20
4	40	41	39	47	37
4	80	79	76	80	76
4	200	193	193	191	191

HMX IN WATER

RUNS 1 2 3 4

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
8.00	7.50	7.50	7.50	7.50
20.00	19.00	19.00	20.00	20.00
40.00	41.00	39.00	40.00	37.00
80.00	79.00	76.00	80.00	76.00
200.00	193.00	193.00	191.00	191.00

HMX IN WATER

RUNS 1 2 3 4

TARGET CONC

ANALYSIS OF 20 TARGET CONC-FOUND CONC POINTS

MEAN= 69.6 SD= 71.4411056672

FOUND CONC

MEAN= 67.2 SD= 68.5389631792

N0. RUNS 4 TOTAL X-Y ALL RUNS 20 N0. CONCENTR 20

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.438753959873

SLOPE= 0.959213305174

R= 0.999829234549

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 1.69335914584

COMPUTE T

D.F.= 18

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 1.73406096408

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 2.80536084237

x(d)= 4.92681190763

HMX IN WATER  
RUNS 1 2 3 4  
FOUND CONC

193.00

y(c)  
x(d)

0.00

40

80

120

160

200

TARGET CONC

VERTICAL AXIS TIC INTERVAL= 19.3

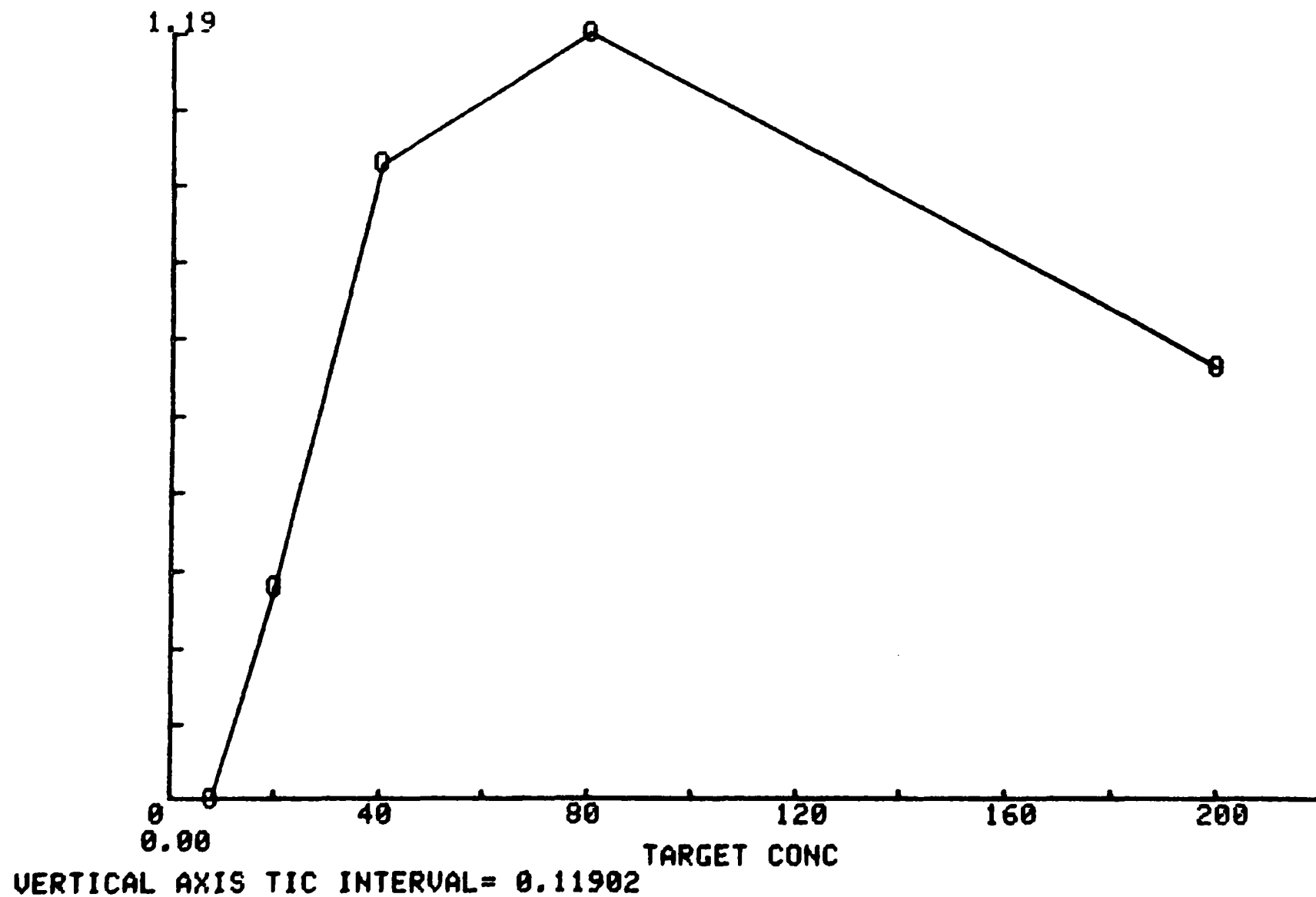
HMX IN WATER

RUNS 1 2 3 4

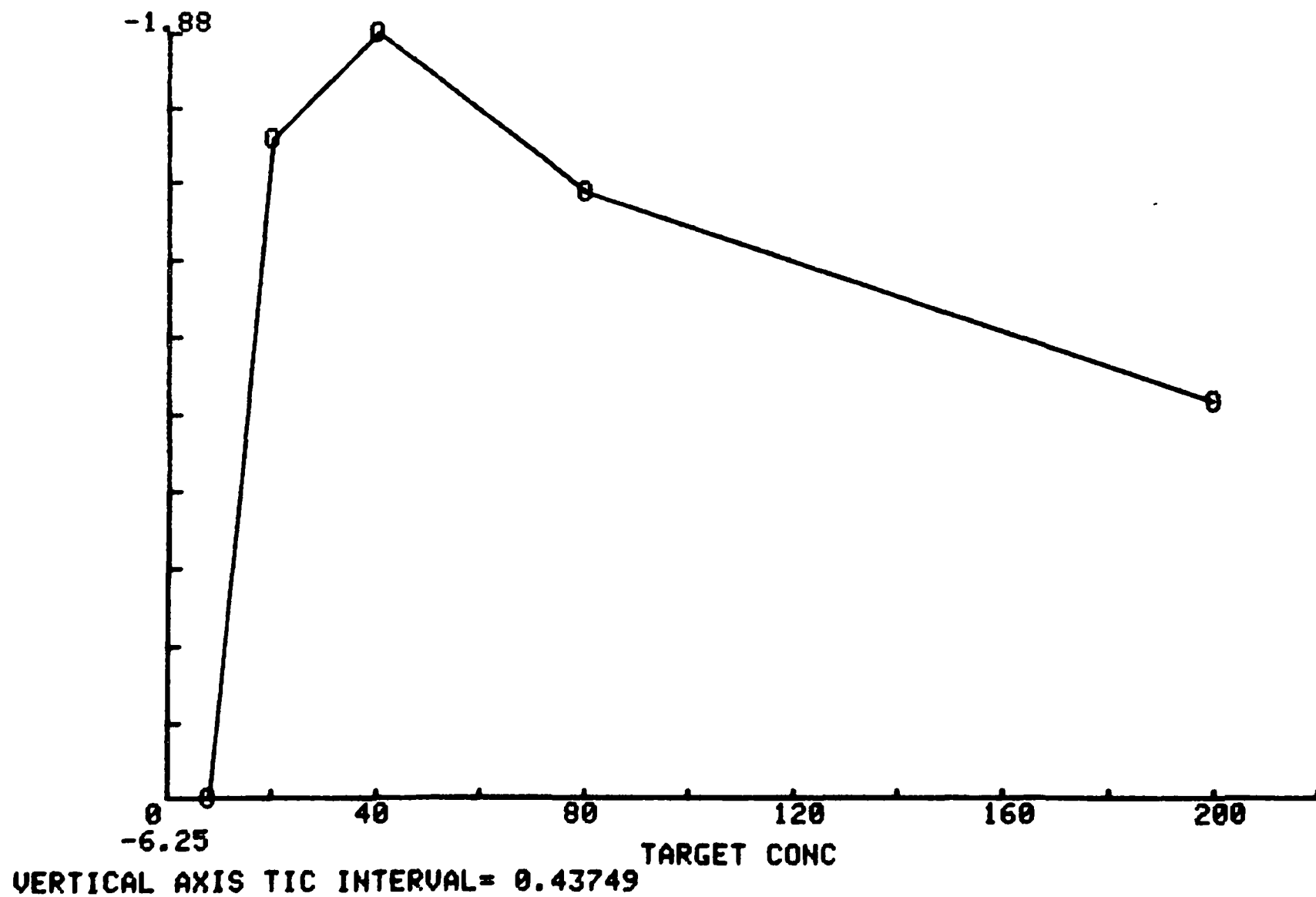
STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION

Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
8.00	7.50	0.00	-6.25	0.00
20.00	19.50	0.33	-2.50	1.71
40.00	39.25	0.99	-1.88	2.51
80.00	77.75	1.19	-2.81	1.53
200.00	192.00	0.67	-4.00	0.35

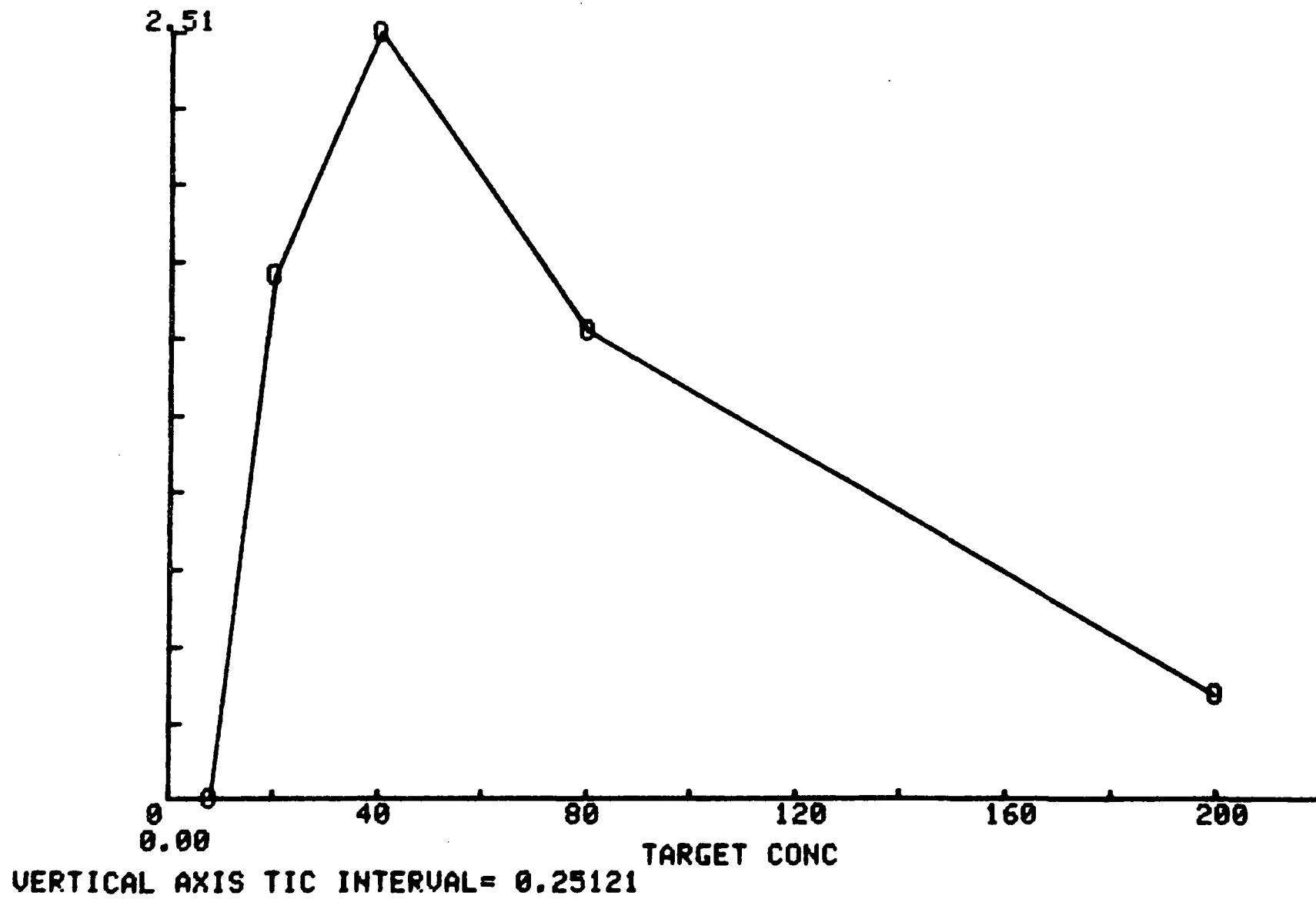
HMX IN WATER  
RUNS 1 2 3 4  
STANDARD DEVIATION



HMX IN WATER  
RUNS 1 2 3 4  
MEAN PERCENT INACCURACY



HMX IN WATER  
RUNS 1 2 3 4  
PERCENT IMPRECISION





THIN LAYER CHROMATOGRAPHIC SCREENING PROCEDURE FOR ORGANIC  
EXPLOSIVES ON BUILDING SURFACES AND IN SOLID PHYSICAL SAMPLES

**I. APPLICATION**

This method is a qualitative and semiquantitative screening procedure for explosives on building surfaces and physical samples.

The method involves 1) extraction of the explosives from the surface or physical sample with acetone, 2) separation of the explosives in the extract by TLC, and 3) identification of the individual components using a sequence of visualization techniques.

- A. Tested Concentration Range. Given below are tested concentration ranges for some representative compounds.

<u>Compound</u>	<u>µg on Plate</u>	<u>Surface Swabs µg/cm<sup>2</sup></u>	<u>Solid Physical samples µg/gram</u>
TNT	3-15	0.4 -2.2	150-170
2,4 DNT	3-15	0.45-2.2	150-170
2,6 DNT	3-15	0.45-2.2	150-750
Tetryl	3-15	0.45-2.2	150-750
Styphnic acid	3-15	0.45-2.2	150-750
NG	4-15	0.6 -2.2	200-750
PETN	4-15	0.6 -2.2	200-750
NC	4-15	0.6 -2.2	200-750

- B. Sensitivity. N/A

- C. Detection Limits. Approximate detection limits for the various compounds are given below.

<u>Compound</u>	<u>µg on Plate</u>	<u>Surface Swabs µg/cm<sup>2</sup></u>	<u>Solid Physical samples µg/gram</u>
TNT	3	0.45	150
2,4 DNT	3	0.45	150
2,6 DNT	8	1.2	400
Tetryl	3	0.45	150
Styphnic acid	3	0.45	150
NG	4	0.6	200
PETN	4	0.6	200
NC	4	0.6	200

- D. Interferences. Unknown compounds can interfere if they have the same RF value as the compound of interest. Use of a variety of visualization methods helps to reduce the number of interferences.
- E. Analysis Rate. Approximately twenty samples can be analyzed by a worker in an eight hour day.

## II. CHEMISTRY

Since this method is designed to be a general screening procedure for explosives individual compounds will not be discussed. In addition to the TLC separation system a series of visualization techniques are used to identify individual compounds. The utility of these visualization techniques is described below.

<u>Visualization Techniques</u>	<u>Comments</u>
Visible light	Colored compounds such as styphnic acid are detected.
UV light	UV active compounds will appear as dark spots on a green background by quenching the fluorescence of the dye on the TLC plate.
10% KOH	TNT for a bright red complex when base is applied. Tetryl also yields a red color.
Geiss reagent	Nitrate esters will give a red color with this spray.

## III. APPARATUS

- A. Instrumentation - N/A
- B. Parameters - N/A
- C. Hardware/Glassware.
1. 2 dram vials with polyseal caps.
  2. TLC tank to hold 20 centimeter plates.
  3. 100- $\mu$ l syringe.
  4. Cotton swabs (Q-tips).
  5. 15- $\mu$ l screw capped centrifuge tubes.
  6. TLC sprayer.

2. Diluted Stock. Ten milliliters of the stock solution are pipetted into a 100 ml volumetric flask. The flask is diluted to the mark with acetone, and its contents are mixed well.
  3. Working. Five calibration standards are prepared from the stock solutions in distilled water by placing 0, 25, 100, 250, 500, and 1000 microliters of diluted stock in a 100 ml volumetric flask and diluting to the mark, to give solutions containing 0, 8, 20, 50, 100, and 200  $\mu\text{g}/\ell$ .
- B. Control Spikes. Control spikes are equivalent to the working standards, since the method involves direct aqueous injection.

## V. PROCEDURE

- A. Sample Handling/Preparation. Filter the samples through a 0.45 micron Millipore filter (Type GC). The samples, in Teflon lined, screw capped, two dram vials are stored in a refrigerator until analyzed.
- B. Calibration. Inject 100  $\mu\text{l}$ s of each standard two times onto the chromatographic system. A solvent blank, represented by distilled water is also analyzed. Record the concentration, retention time and peak height for HMX. Average the peaks heights for the duplicate injections of each standard.
- C. Analysis. Inject 100  $\mu\text{l}$  of each sample onto the chromatographic system. Record the retention time and peak height for peaks eluting at the same time as HMX (within 0.1 minute of the HMX standard).

## VI. CALCULATIONS

A linear regression curve (concentration vs. peak height) is generated from the HMX calibration data. The sample concentrations are interpolated using the curve.

The relationship between found component concentration and sample volume of water is as follows:

$$\text{ng found} \div 100 \mu\text{l} = \frac{\text{Conc}(\mu\text{g}/\text{l})}{1000}$$

square centimeter areas within the marked area. Place the swabs in the vial, cap tightly, and return to the laboratory for analysis.

(2) Physical samples. Place 100 milligrams of the residue in a screw capped centrifuge tube containing 0.5 milliliters of acetone. Shake gently for 1 minute and let stand overnight. Remove a portion of the acetone extract for analysis.

- B. TLC Separation. Spot 100 microliters of the acetone extract on the TLC plate. Spot standard explosives on separate channels at various levels (1-10 micrograms for most compounds). Allow the spots to dry on the plate.

Place the plate in a covered TLC tank lined with filter paper and containing 100 ml of 15% butyl acetate/85% petroleum ether (equilibrated 1 hour before placing the plate in the tank).

When the solvent front has advanced -15 centimeters remove the plate from the tank, mark the solvent front and allow the plate to air dry in a hood.

- C. Visualization and Component Identification. Apply the following sequence of visualization techniques:

- Visible light
- UV light
- Spray with 10% KOH
- Heat in oven at 105°C for 30 minutes
- Spray with modified Griess reagent.

Note all spots and measure their RF values. If the RF value of a particular component matches one of the standards estimate the level by visual comparison of the spot intensities of the sample and standard.

If a component responds equivocally to one of the standards in all visualization methods and has an RF value within .05 units of the expected value it should be reported as the compound of interest.

# VI. CALCULATIONS

$$RF = \frac{\text{Distance travelled by component (cm)}}{\text{Distance travelled by solvent front (cm)}}$$

For building surfaces:

$$\text{concentration } (\mu\text{g}/\text{cm}^2) = \mu\text{g}/\text{plate} \times 0.15$$

For physical samples

$$\text{concentration } (\mu\text{g}/\text{gram}) = \mu\text{g}/\text{plate} \times 50$$

# VII. REFERENCES    None

# VIII. SUPPORTING DATA

The following table provides typical RF and response data for several explosives of interest.

Compound	RF	Visible	UV	10% KOH	Griess
TNT	0.83		+	red color	
2,4 DNT	0.63		+		
2,6 DNT	0.73		+		
Tetryl	0.44			brown-red color	
Styphnic acid	0	yellow color	+		
NG	0.64				red color
PETN	0.82				red color
NC	0				red color

The accuracy of this method has been determined at various levels for several explosives by running a series of blind acetone spikes. These data are presented in the following table:

Compound	<u>Concentration (ug on plate)</u>									
	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found	Spiked	Found
TNT	0	0	3	5	6	10	8	15	15	13
2,4 DNT	0	0	3	4	6	5	8	10	15	12
2,6 DNT	0	0	3	ND	6	ND	8	4	15	6
Tetryl	0	0	3	3	6	4	8	11	15	18
Styphnic acid	0	0	3	7	6	23	8	12	15	33
NC	0	0	4	18	8	25	10	37	15	30
PETN	0	0	4	8	8	12	10	22	15	17
NC	0	0	4	2	8	5	10	7	15	7

The precision of the method is estimated to be  $\pm 25\%$ , on the basis of intensities observed for replicate standards. The precision is highly dependant on the person conducting the analysis as well as the sample background level.

DETERMINATION OF 2,4-DNT, 2,6-DNT, TNT AND  
TETRYL IN WATER AND SOIL

I. Application: This method is used to qualitatively determine the concentration of 2,4-DNT, 2,6-DNT, TNT, and tetryl in water and soil samples.

A. Tested Concentration Range - The concentration ranges are as follows:

	<u>Water</u>	<u>Soil</u>
2,6-DNT	0.1-10	--
2,4-DNT	0.2-8.1	0.51-5.1
TNT	0.16-6.7	0.42-4.2
Tetryl	0.2-8.1	0.51-5.1

B. Sensitivity Limits -

At the detection limit, 1 ng of 2,6-DNT = 1,150,000 area counts  
1 ng of 2,4-DNT = 1,150,000 area counts  
1 ng of TNT = 1,060,000 area counts  
1 ng of Tetryl = 378,000 area counts

C. Detection Limits - (Calculated using the method of Hubaux and Vox).

In water:

2,6-DNT	0.64 µg/l
2,4-DNT	0.39 µg/l
TNT	1.5 µg/l
Tetryl	1.3 µg/l

In soil:

2,4-DNT	0.51 µg/g (lowest std)
TNT	1.9 µg/g
Tetryl	1.1 µg/g

D. Interferences - None observed for water samples. Several soil samples had a large, broad eluting peak which interfered with the determination of 2,4-DNT, 2,6-DNT, and TNT. In these cases, the detection limit was reduced by a factor of 10, due to the sample dilution required.

E. Analysis Rate - Approximately 10 water samples or 15 soil samples can be analyzed by one worker in an eight hour day.

## II. Chemistry

$C_7H_6N_2O_4$  Toluene, 2,4-Dinitro-

CAS RN 121-14-2

Melting Pt-71C Boiling Pt-300 C (decomposes)

$C_7H_6N_2O_4$  Toluene, 2,6-Dinitro-

CAS RN 606-20-2

Melting Pt-66 C

$C_7H_5N_3O_6$  Toluene, 2,4,6-Trinitro-

CAS RN 118-96-7

Melting Pt-82 C Boiling Pt-240 C (explodes)

$C_7H_5N_5O_8$  Aniline, N-Methyl-N,2,4,6-Tetranitro-

CAS RN 479-45-8

Melting Pt-131 C Boiling Pt-187 C (explodes)

Hazards: Use caution in handling. Potential explosive, toxic inhalation, and skin absorption hazards exist.

### A. Chemical Reactions - N/A

### B. Retention Times

2,6-DNT	2.80 minutes
2,4-DNT	3.60 minutes
TNT	8.65 minutes
Tetryl	18.40 minutes

### C. Hardware/Glassware

1. 1000 ml separatory funnels
2. 2 dram vials with polyseal caps
3. 5 ml volumetric flasks
4. 250 ml bottles with polyseal caps
5. 100 ml pipet
6. refrigerator
7. shaker
8. 30 mesh sieve
9. riffler
10. Kuderna-Danish tubes and columns
11. heated water bath
12. 100 ml volumetric flask
13. Millipore All Glass Filter Apparatus
14. Millipore Filters, 0.45 micron, type HA, 47 mm diameter

#### D. Chemicals

1. benzene, DIG grade, Burdick and Jackson
2. acetone, DIG grade, Burdick and Jackson

E. Reagents None.

### III. Standards

- A. Stock - Weigh 10 mg each of 2,4-DNT, 2,6-DNT, TNT, and Tetryl into a 100 ml volumetric flask, dilute to the mark with benzene and mix thoroughly.
- B. Working - Six calibration standards are prepared from the stock solutions to give solutions containing each component at levels of 0.02, 0.05, 0.1, 0.2, 0.5, and 1.0  $\mu\text{g/ml}$  by placing 20, 50, 100, 200, 500, and 1000  $\mu\text{l}$  of stock in a 100 ml flask and diluting to the mark with benzene.

### IV. Procedure

#### A. Water Samples/Control Spikes

1. Sample Handling/Preparation - Refrigerate the samples until they are to be extracted. Filter through a 0.45 micron Millipore filter prior to analysis.
2. Extraction - Measure 500 ml of the sample into the separatory funnel. Add 25 ml of benzene and shake vigorously. Allow the layers to separate and remove the benzene layer. Transfer to a Kuderna-Danish tube with a short distillation column and concentrate the benzene to less than 5 ml using a heated water bath in a well ventilated hood. Transfer the benzene to a 5 ml volumetric flask and dilute to the mark with benzene.
3. Calibration - Inject 2  $\mu\text{l}$  of each calibration standard in duplicate onto the GC column. Record the retention time, concentration, and peak area for each component.
4. Analysis - Inject 2  $\mu\text{l}$  of sample extract onto the GC column. Record the retention time and peak area for the peaks of interest.

#### B. Soils

1. Sample Handling/Preparation - Samples are air dried and then sieved through a 30 mesh screen. Particles passing through are retained for analysis. Samples are then riffled to obtain even particle size distribution.

2. Extraction - Weigh 10 g of sample into a 250 ml bottle. Add 100 ml of DIG acetone and shake for 1 hour using a mechanical shaker. Allow particles to settle for 1 hour and then transfer an aliquot to a 2 dram vial. Refrigerate until ready to analyze.
3. Calibration - Inject 2  $\mu$ l of each standard onto the GC column. Record the retention time, concentration, and peak area for each component.
4. Analysis - Inject 2  $\mu$ l of each sample onto the GC column. Record the retention time and peak areas for those peaks of interest.

#### V. Calculations

Use the calibration data from each component to establish a linear regression curve for each component. Interpolate sample concentration from these curves.

The relationship between found component concentration and sample volume of water is as follows:

$$\text{ng}/2 \mu\text{l} = \mu\text{g}/5\text{ml benzene} \div 2.5 = \mu\text{g}/500 \text{ ml water} \div 2.5$$

$$\text{ng}/2 \mu\text{l} \times 5 = \mu\text{g}/\text{liter} = \text{ppb}$$

The relationship between found component concentration and sample weight for soil is as follows:

$$\text{ng}/2 \mu\text{l} = \frac{\mu\text{g in sample}}{50}$$

$$\mu\text{g in sample} = \text{ng}/2 \mu\text{l} \times 50$$

$$\frac{\mu\text{g found}}{\text{sample wt. (gram)}} = \text{ppm}$$

#### VI. References - None.

2,6-DNT IN WATER

1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
0.50	0.42			
1.00	0.75			
2.00	2.00			
5.00	5.10			

2,6-DNT IN WATER

1

TARGET CONC

ANALYSIS OF 5 TARGET CONC-FOUND CONC POINTS

MEAN= 1.7 SD= 1.98746069144

FOUND CONC

MEAN= 1.654 SD= 2.06581218895

N0. RUNS 1 TOTAL X-Y ALL RUNS 5 N0. CONCENTR 5

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.110664556962

SLOPE= 1.03803797468

R= 0.998667585533

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0151530590719

COMPUTE T

D.F.= 3

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.3533525706

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.230005789158

x(d)= 0.642785953083



2,6-DNT IN WATER

1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00			
0.50	0.42			
1.00	0.75			
2.00	2.00			
5.00	5.10			
10.00	8.80			

2,6-DNT IN WATER

1

TARGET CONC

ANALYSIS OF 6 TARGET CONC-FOUND CONC POINTS

MEAN= 3.08333333333 SD= 3.8264430829

FOUND CONC

MEAN= 2.845 SD= 3.45325209042

N0. RUNS 1 TOTAL X-Y ALL RUNS 6 N0. CONCENTR 6

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0725270347183

SLOPE= 0.899180421172

R= 0.996354338688

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.108487706318

COMPUTE T

D.F.= 4

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 2.13183865604

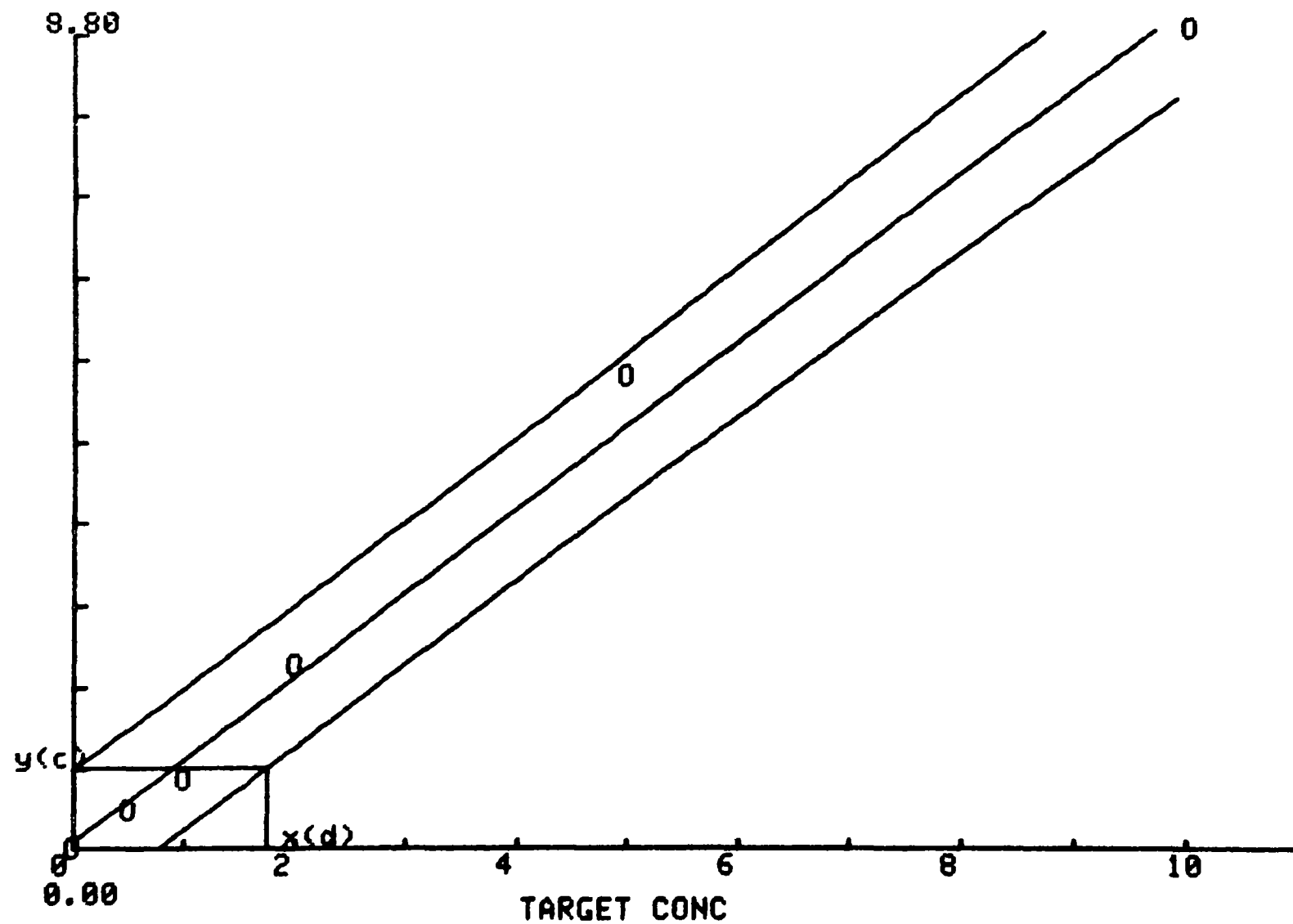
REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.872058528967

x(d)= 1.7415015287

2,6-DNT IN WATER

1  
FOUND CONC



VERTICAL AXIS TIC INTERVAL = 0.88

2,4-DNT IN WATER

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.00	0.00	0.00	0.00	
0.20	0.15	0.20	0.17	
0.51	0.45	0.47	0.42	
1.02	0.85	0.68	1.05	

3,4-DNT IN WATER

RUNS 1 2 3

TARGET CONC

ANALYSIS OF 12 TARGET CONC-FOUND CONC POINTS

MEAN= 0.4325 SD= 0.401907384175

FOUND CONC

MEAN= 0.37 SD= 0.348477206767

N0. RUNS 3 TOTAL X-Y ALL RUNS 12 N0. CONCENTR 12

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.00519888002476

SLOPE= 0.843470797631

R= 0.972795739063

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00716903155911

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

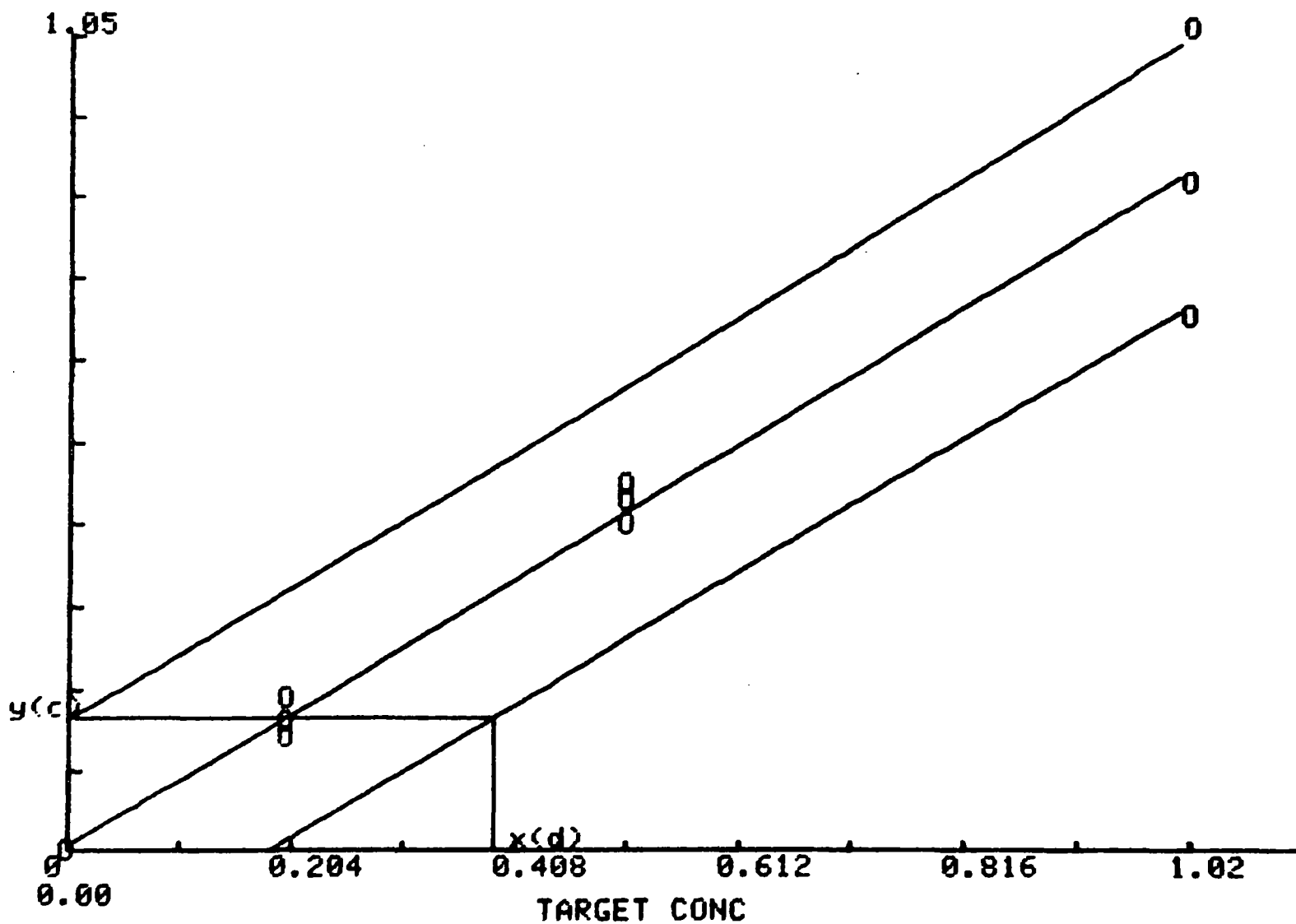
t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.172507320123

x(d)= 0.387824637321

2,4-DNT IN WATER  
 RUNS 1 2 3  
 FOUND CONC



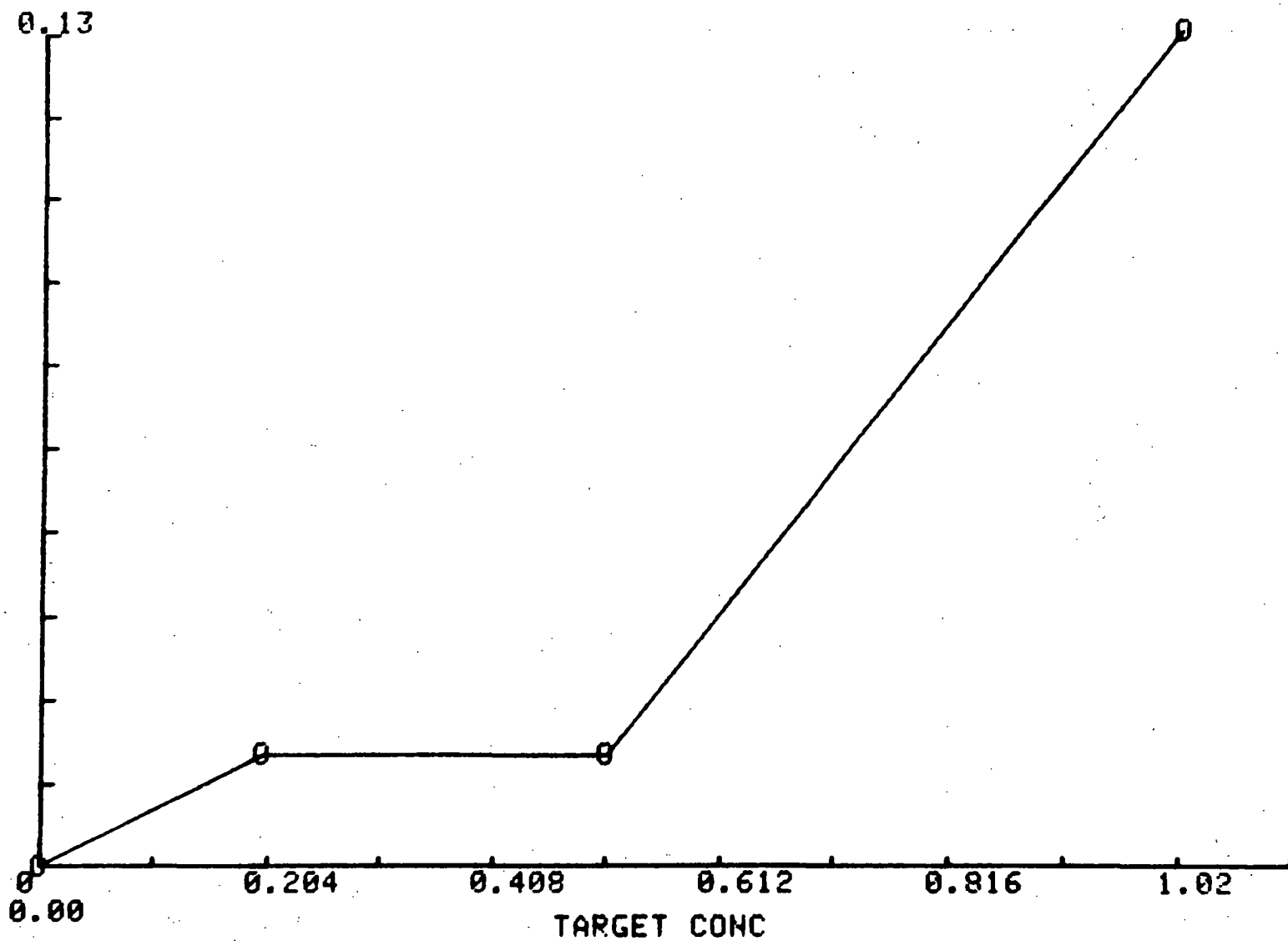
2,4-DNT IN WATER

RUNS 1 2 3

STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION

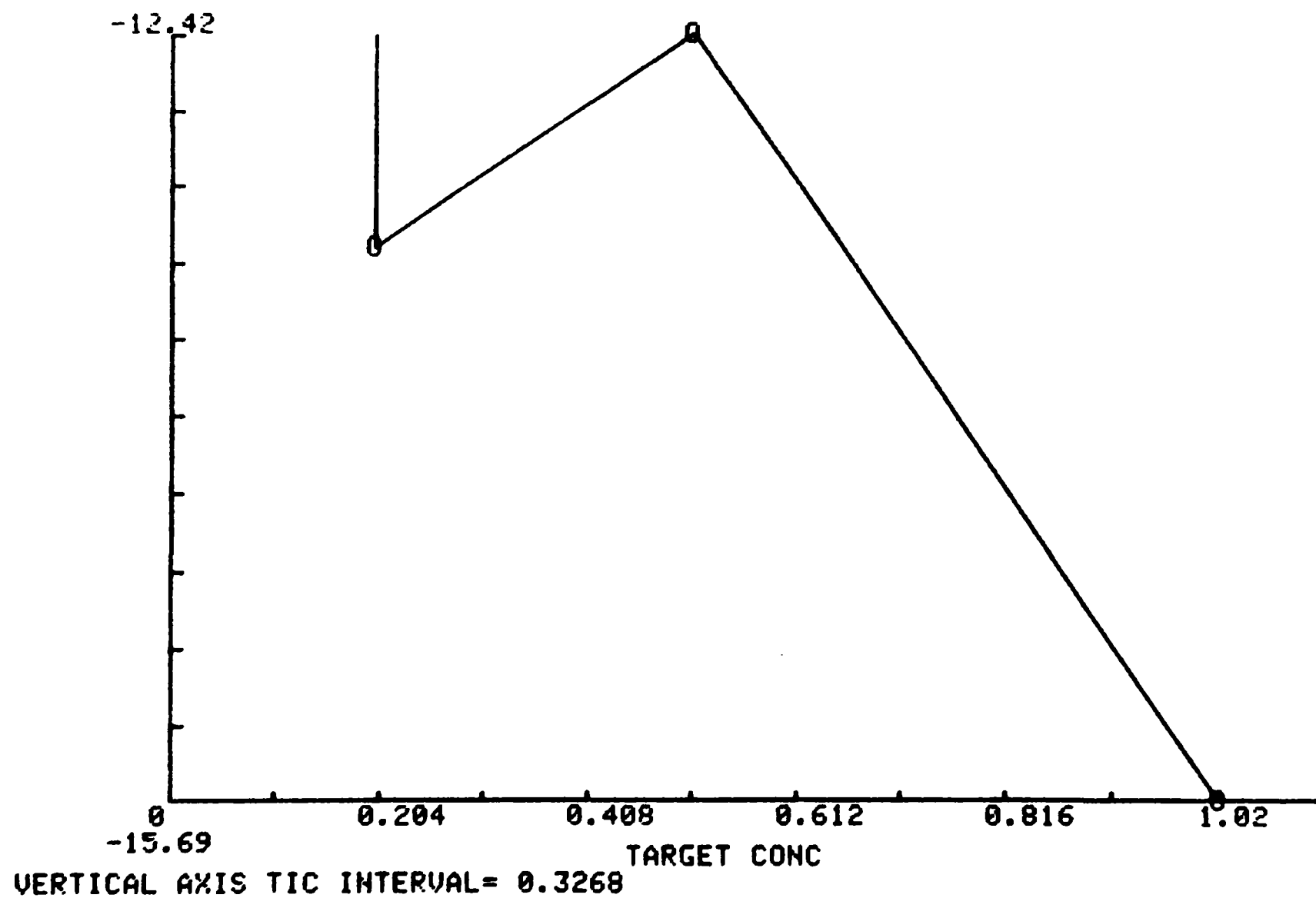
Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.00	0.00	0.00		
0.20	0.17	0.02	-13.33	10.27
0.51	0.45	0.02	-12.42	3.98
1.02	0.86	0.13	-15.69	15.23

2,4-DNT IN WATER  
RUNS 1 2 3  
STANDARD DEVIATION

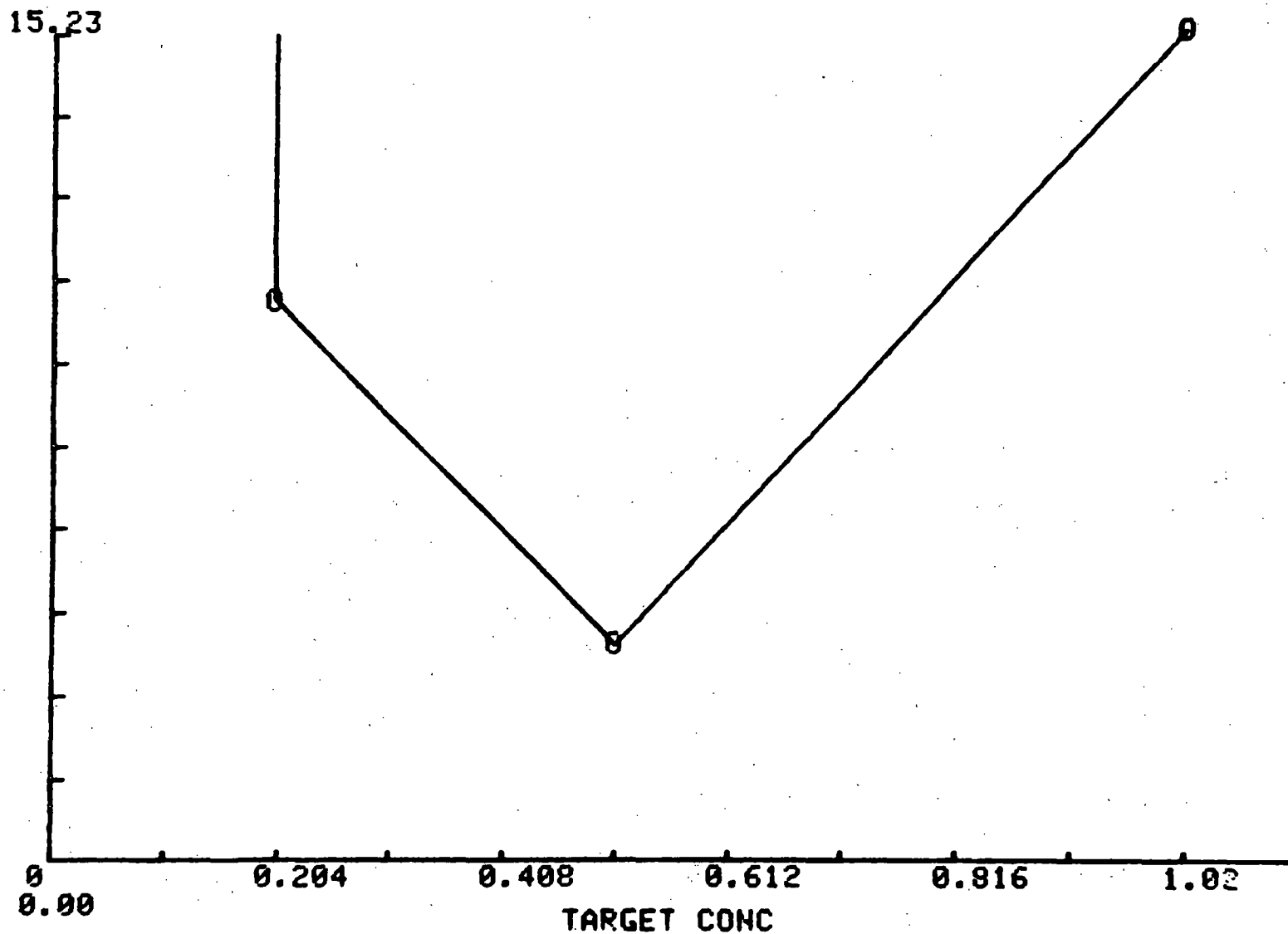


VERTICAL AXIS TIC INTERVAL= 0.01309

2,4-DNT IN WATER  
PUNS 1 2 3  
MEAN PERCENT INACCURACY



2,4-DNT IN WATER  
RUNS 1 2 3  
PERCENT IMPRECISION



VERTICAL AXIS TIC INTERVAL= 1.52276

2,4-DNT IN WATER

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.20	0.15	0.20	0.17	
0.51	0.45	0.47	0.42	
1.02	0.85	0.68	1.05	
2.04	1.37	1.43	1.90	
4.08	3.63	3.55	3.79	
8.16	7.26	7.13	7.83	

2,4-DHT IN WATER

RUNS 1 2 3

TARGET CONC

ANALYSIS OF 18 TARGET CONC-FOUND CONC POINTS

MEAN= 2.669 SD= 2.84897349935

FOUND CONC

MEAN= 2.35166666667 SD= 2.61175228534

NØ. RUNS 3 TOTAL X-Y ALL RUNS 18 NØ. CONCENTR 18  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.087540182218

SLOPE= 0.913902903291

R= 0.996911218214

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0447032285582

COMPUTE T

D.F.= 16

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 1.74588066418

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.30087257849

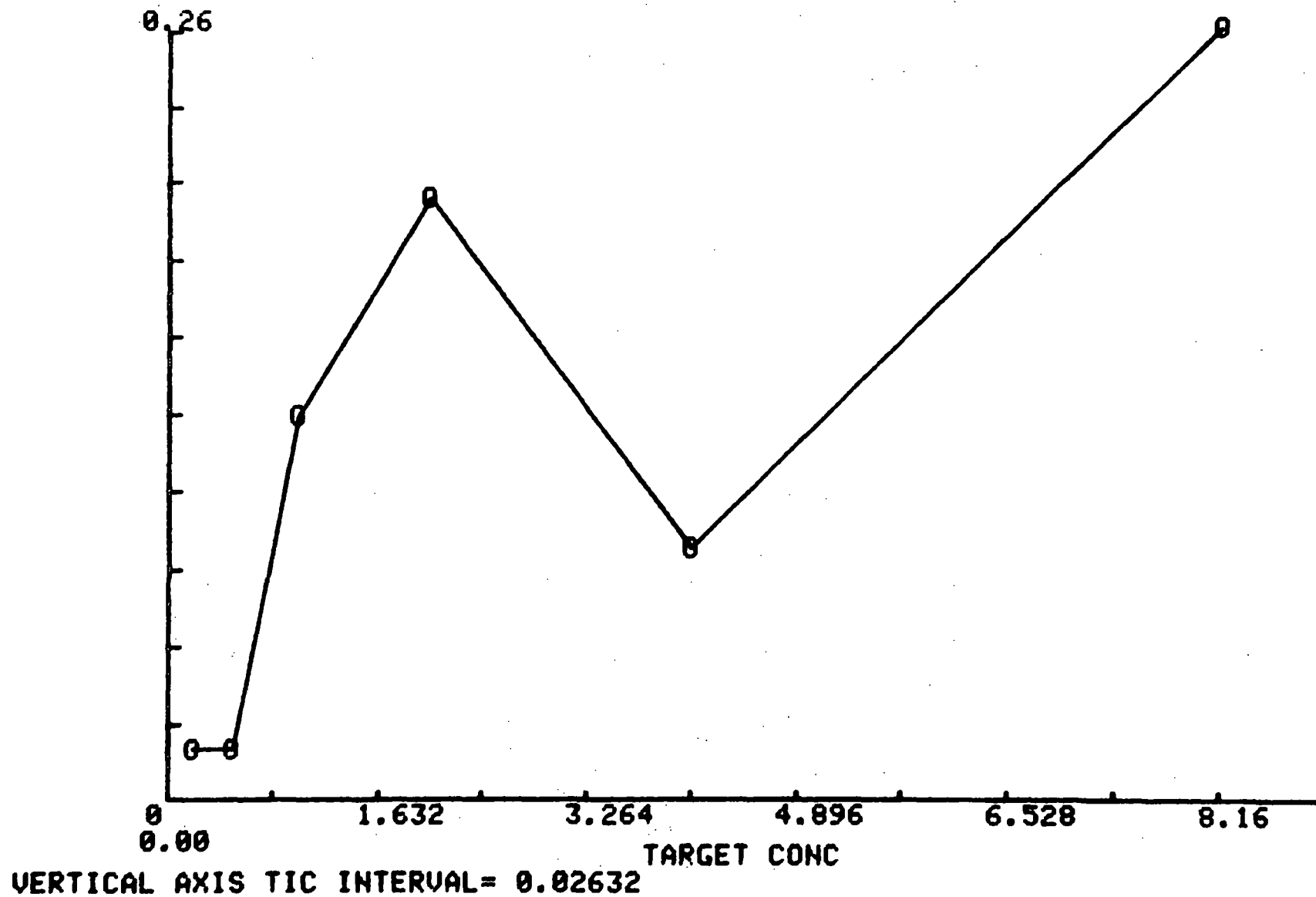
x(d)= 0.844696342542

[illegible]

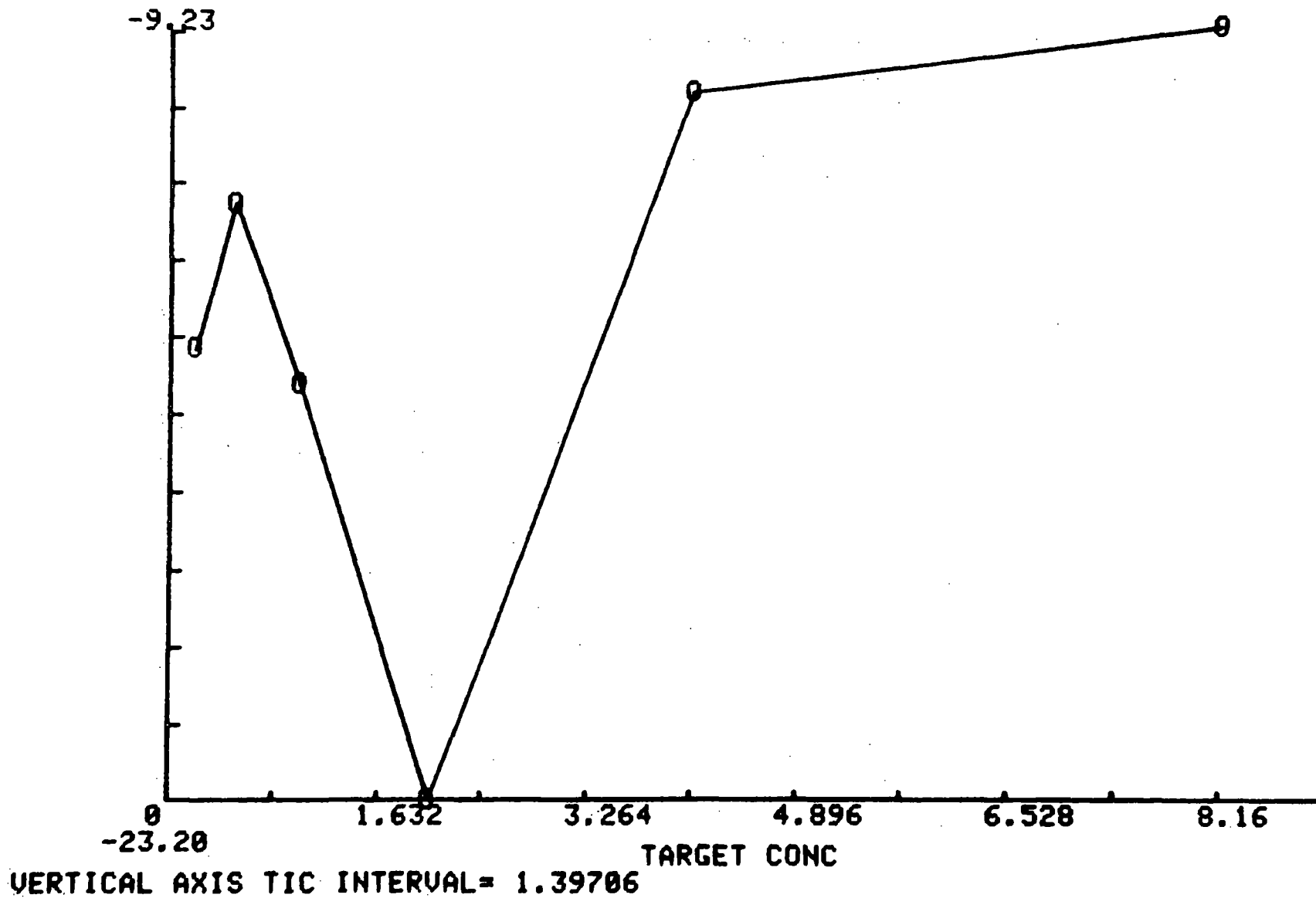
**2,4-DNT IN WATER****RUNS 1 2 3****STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION**

<b>Target Conc. UG/L</b>	<b>Mean Found Conc. UG/L</b>	<b>Standard Deviation</b>	<b>Percent Inaccuracy</b>	<b>Coefficient(%) of variation</b>
0.20	0.17	0.02	-15.03	10.27
0.51	0.45	0.02	-12.42	3.98
1.02	0.86	0.13	-15.69	15.23
2.04	1.57	0.21	-23.20	13.10
4.08	3.66	0.09	-10.38	2.36
8.16	7.41	0.26	-9.23	3.55

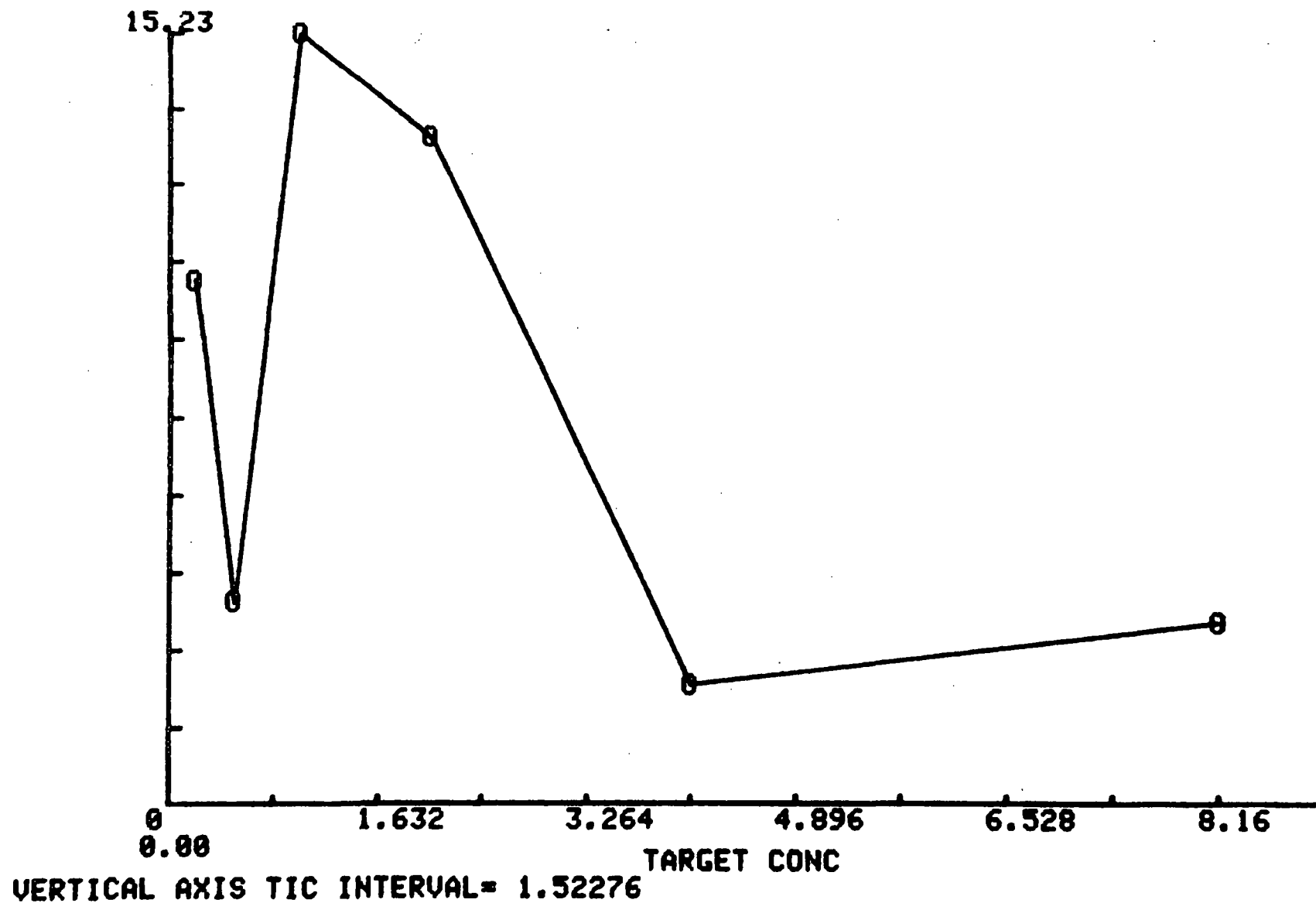
2,4-DNT IN WATER  
RUNS 1 2 3  
STANDARD DEVIATION



2,4-DNT IN WATER  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



2,4-DNT IN WATER  
RUNS 1 2 3  
PERCENT IMPRECISION



**TNT IN WATER**

**RUNS 1 2 3**

**COMPILATION OF TARGET CONC. VS FOUND CONC**

<b>Target Conc. UG/L</b>	<b>Day 1 Found Conc. UG/L</b>	<b>Day 2 Found Conc. UG/L</b>	<b>Day 3 Found Conc. UG/L</b>	<b>Day 4 Found Conc. UG/L</b>
<b>0.17</b>	<b>0.12</b>	<b>0.06</b>	<b>0.12</b>	
<b>0.42</b>	<b>0.32</b>	<b>0.11</b>	<b>0.27</b>	
<b>0.84</b>	<b>0.65</b>	<b>0.18</b>	<b>0.75</b>	
<b>1.68</b>	<b>1.13</b>	<b>0.35</b>	<b>1.46</b>	
<b>3.36</b>	<b>2.99</b>	<b>2.52</b>	<b>3.19</b>	

TNT IN WATER  
RUNS 1 2 3  
TARGET CONC  
ANALYSIS OF 15 TARGET CONC-FOUND CONC POINTS

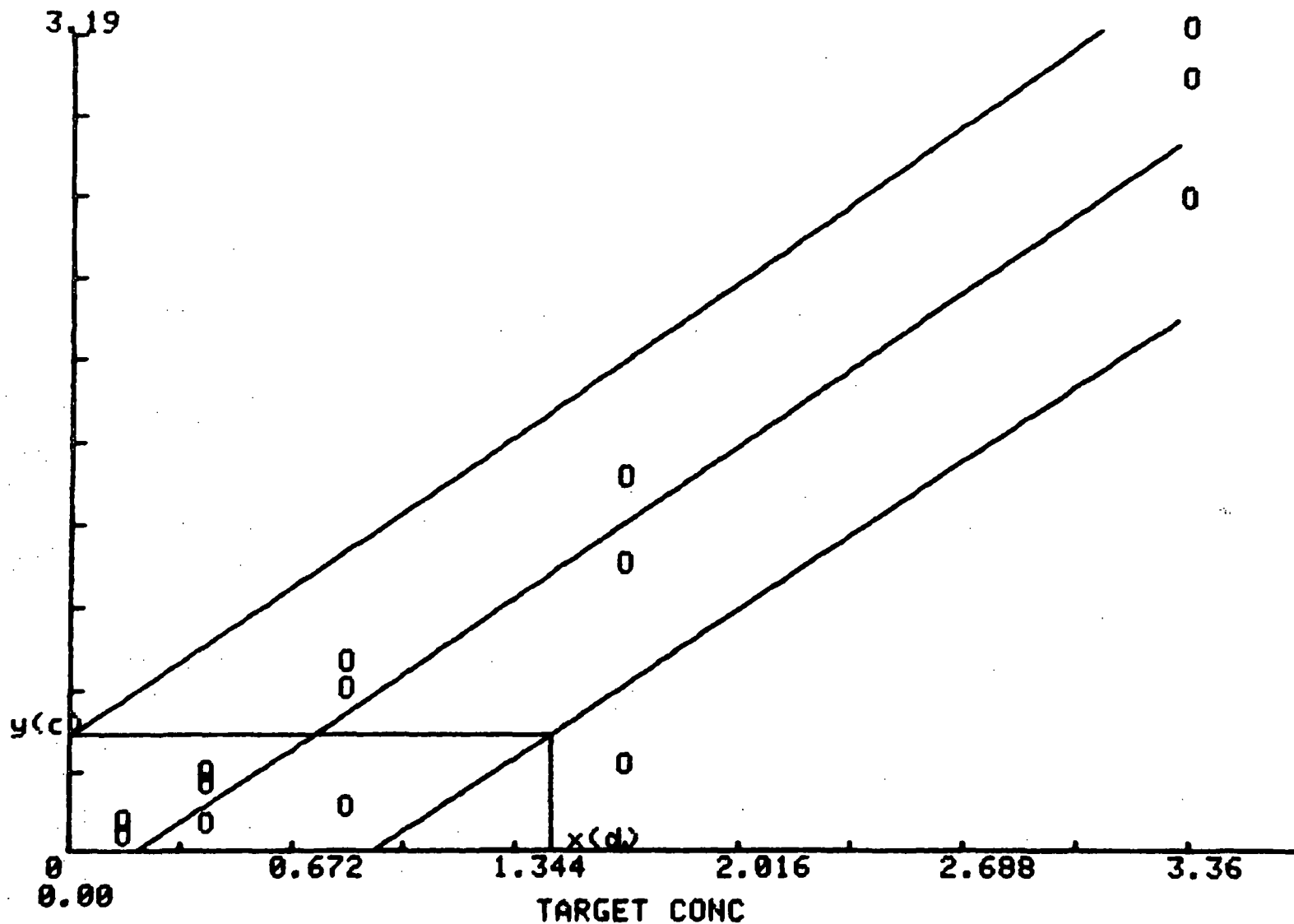
MEAN= 1.2932 SD= 1.19436505546

FOUND CONC  
MEAN= 0.948 SD= 1.0939522058

NO. RUNS 3 TOTAL X-Y ALL RUNS 15 NO. CONCENTR 15  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.181970946192  
SLOPE= 0.873778956227  
R= 0.953982309269  
MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.115884894163  
COMPUTE T  
D.F.= 13  
ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)  
.1  
t= 1.77093170942  
REPLICATES ON UNKNOWN SAMPLE 1  
y(c)= 0.464636625642  
x(d)= 1.45332273315

TNT IN WATER  
 RUNS 1 2 3  
 FOUND CONC



VERTICAL AXIS TIC INTERVAL = 0.319

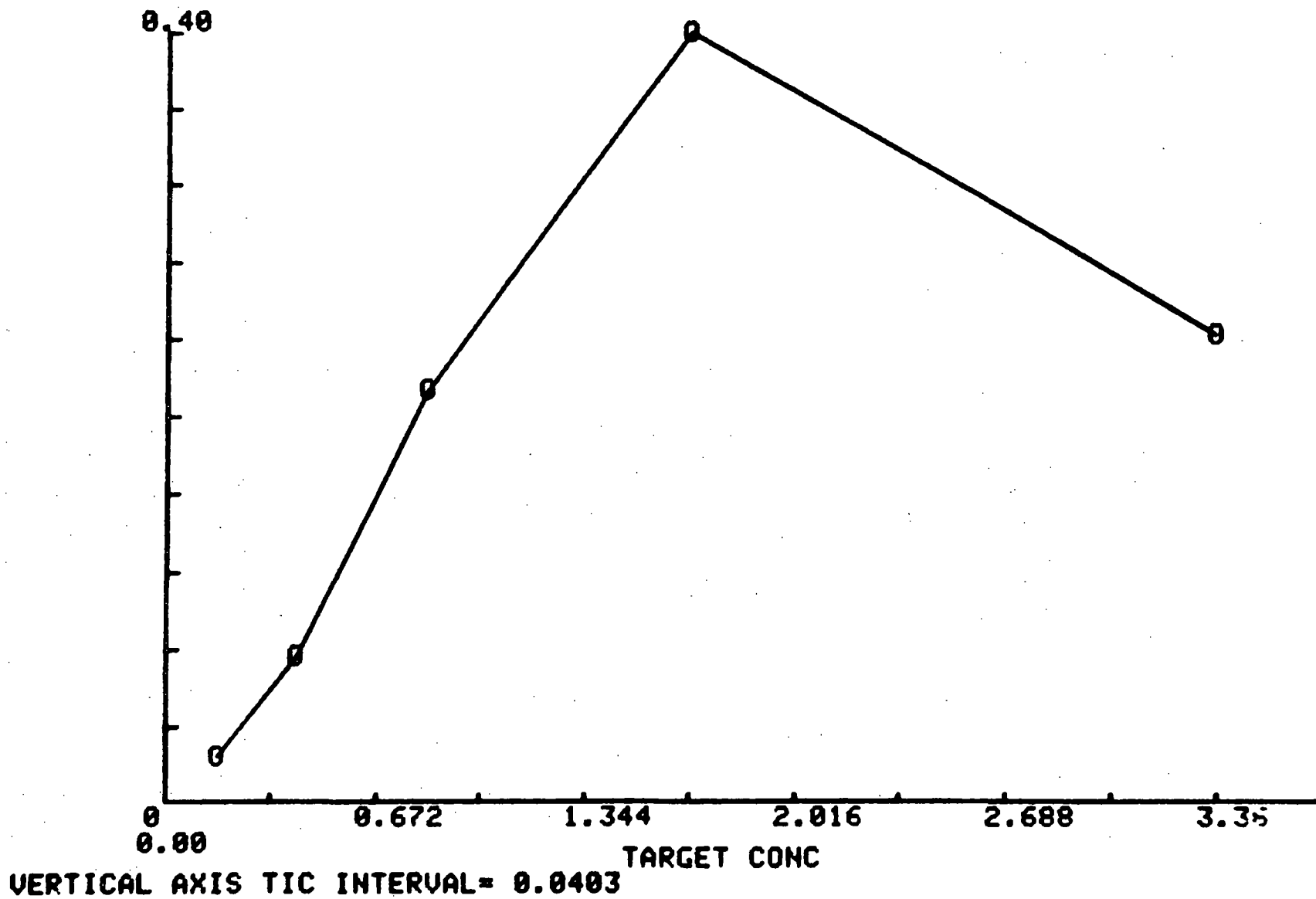
TNT IN WATER

RUNS 1 2 3

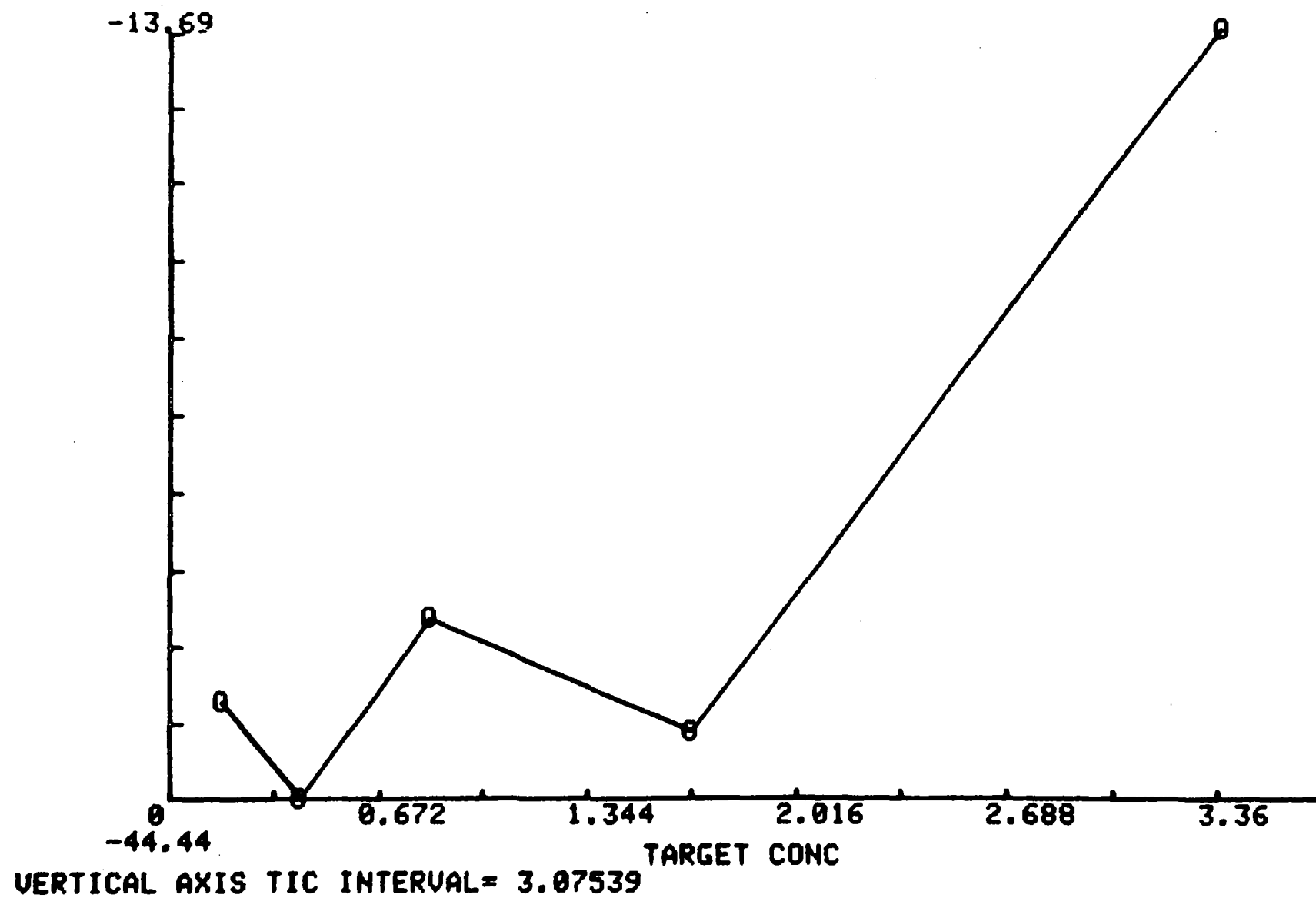
STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION

Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.17	0.10	0.02	-40.48	24.49
0.42	0.23	0.08	-44.44	33.24
0.84	0.53	0.22	-37.15	40.86
1.68	0.98	0.40	-41.67	41.13
3.36	2.90	0.24	-13.69	8.39

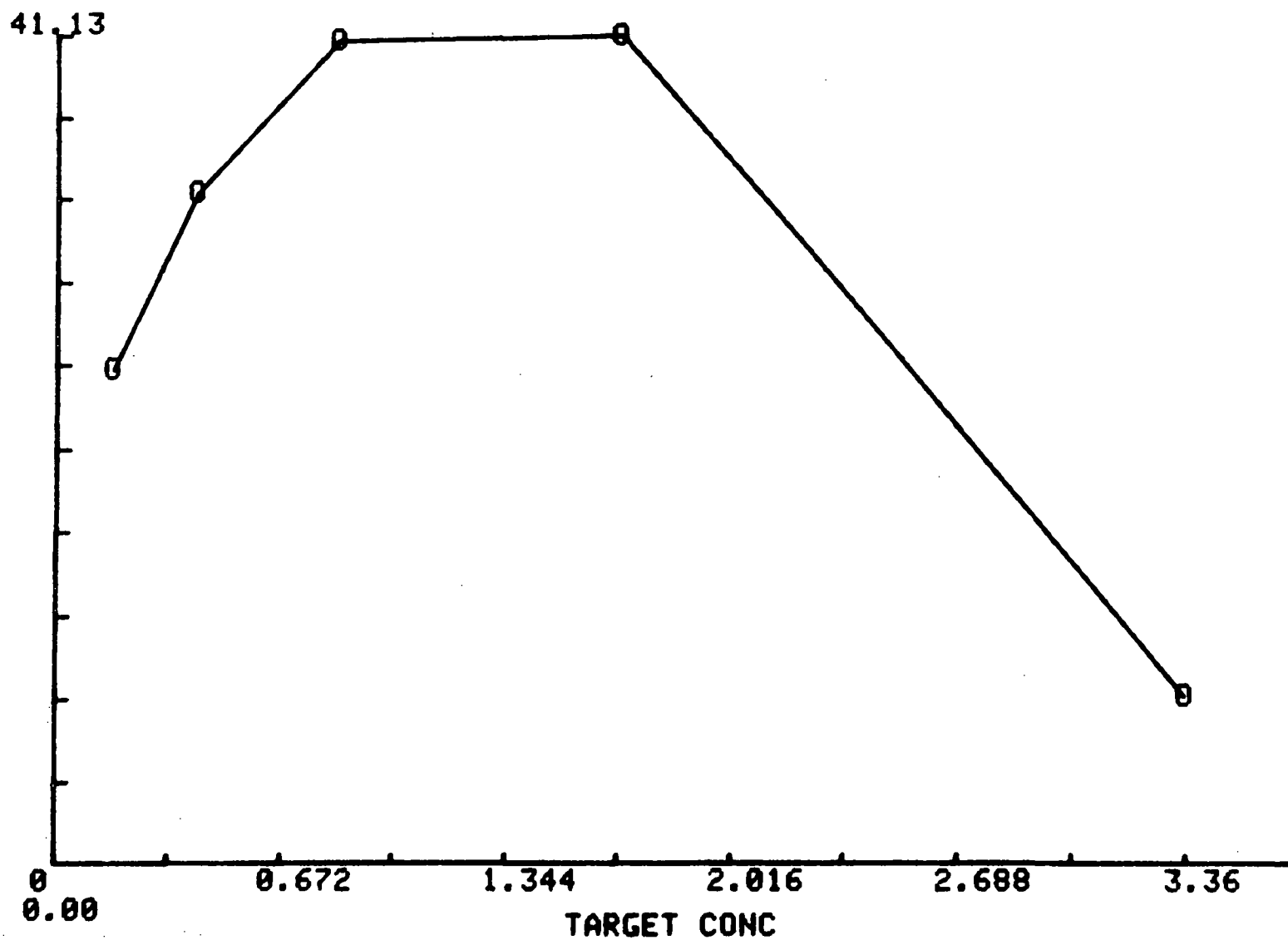
TNT IN WATER  
RUNS 1 2 3  
STANDARD DEVIATION



TNT IN WATER  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



TNT IN WATER  
RUNS 1 2 3  
PERCENT IMPRECISION



VERTICAL AXIS TIC INTERVAL= 4.11276

TNT IN WATER

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.17	0.12	0.06	0.12	
0.42	0.32	0.11	0.27	
0.84	0.65	0.18	0.75	
1.68	1.13	0.35	1.46	
3.36	2.99	2.52	3.19	
6.72	3.91	4.37	3.96	

TNT IN WATER

RUNS 1 2 3

TARGET CONC

ANALYSIS OF 18 TARGET CONC-FOUND CONC POINTS

MEAN= 2.1976666667 SD= 2.34641787061

FOUND CONC

MEAN= 1.47 SD= 1.56064089399

N0. RUNS 3 TOTAL X-Y ALL RUNS 18 N0. CONCENTR 18

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0625443529555

SLOPE= 0.6404318127

R= 0.962886885775

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.188520062837

COMPUTE T

D.F.= 16

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

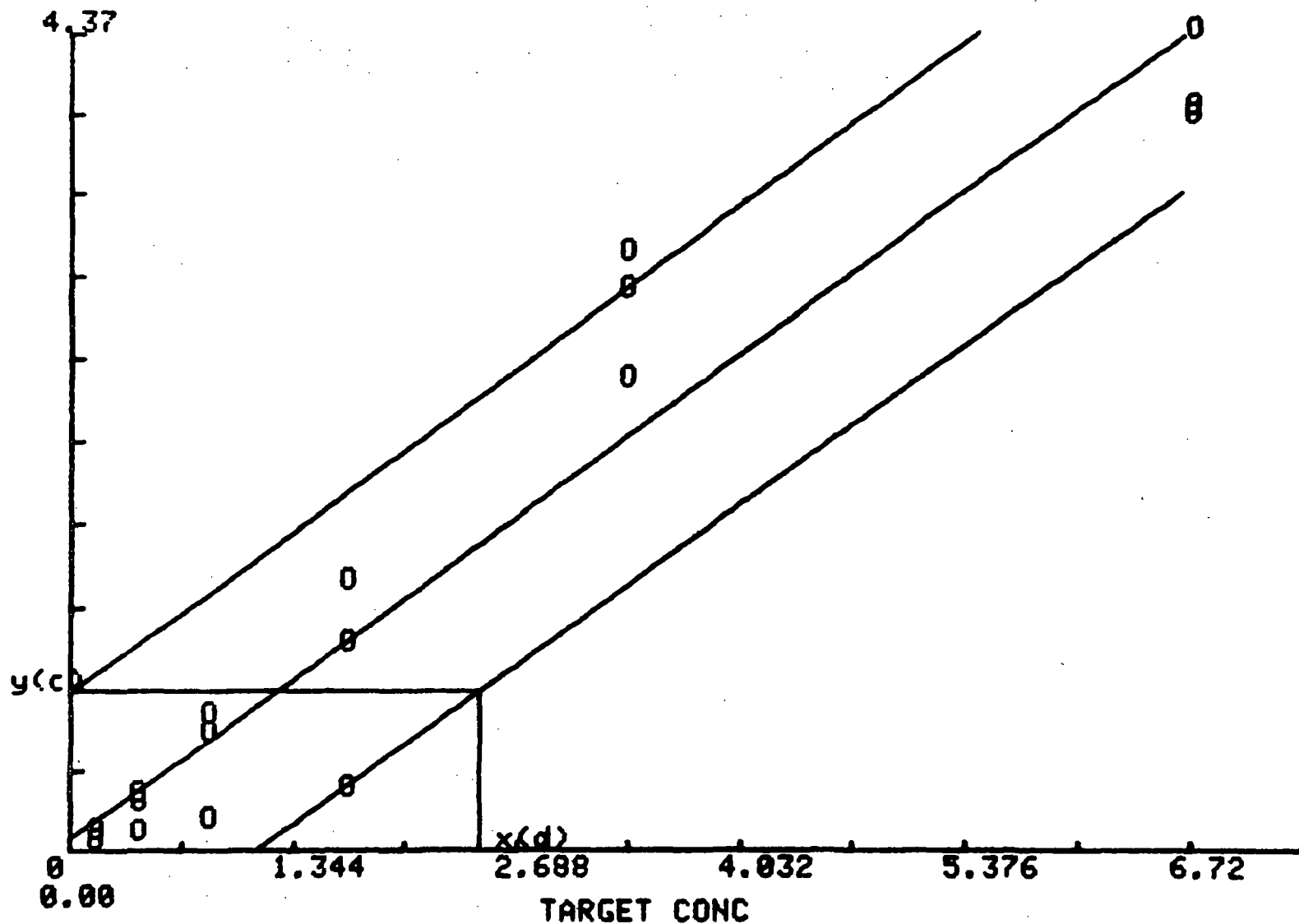
t= 1.74588066418

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.860167958188

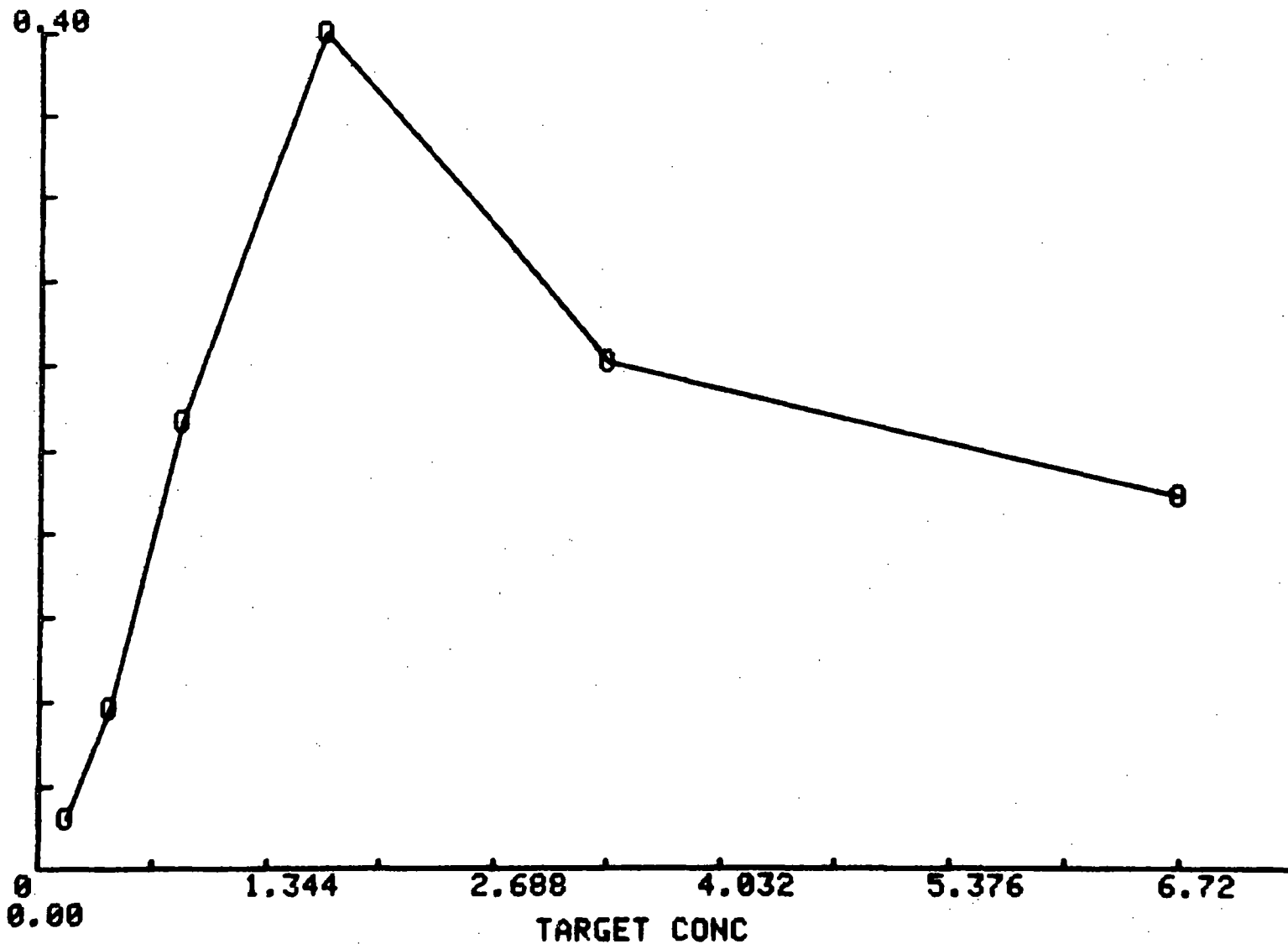
x(d)= 2.46215133926

TNT IN WATER  
 RUNS 1 2 3  
 FOUND CONC



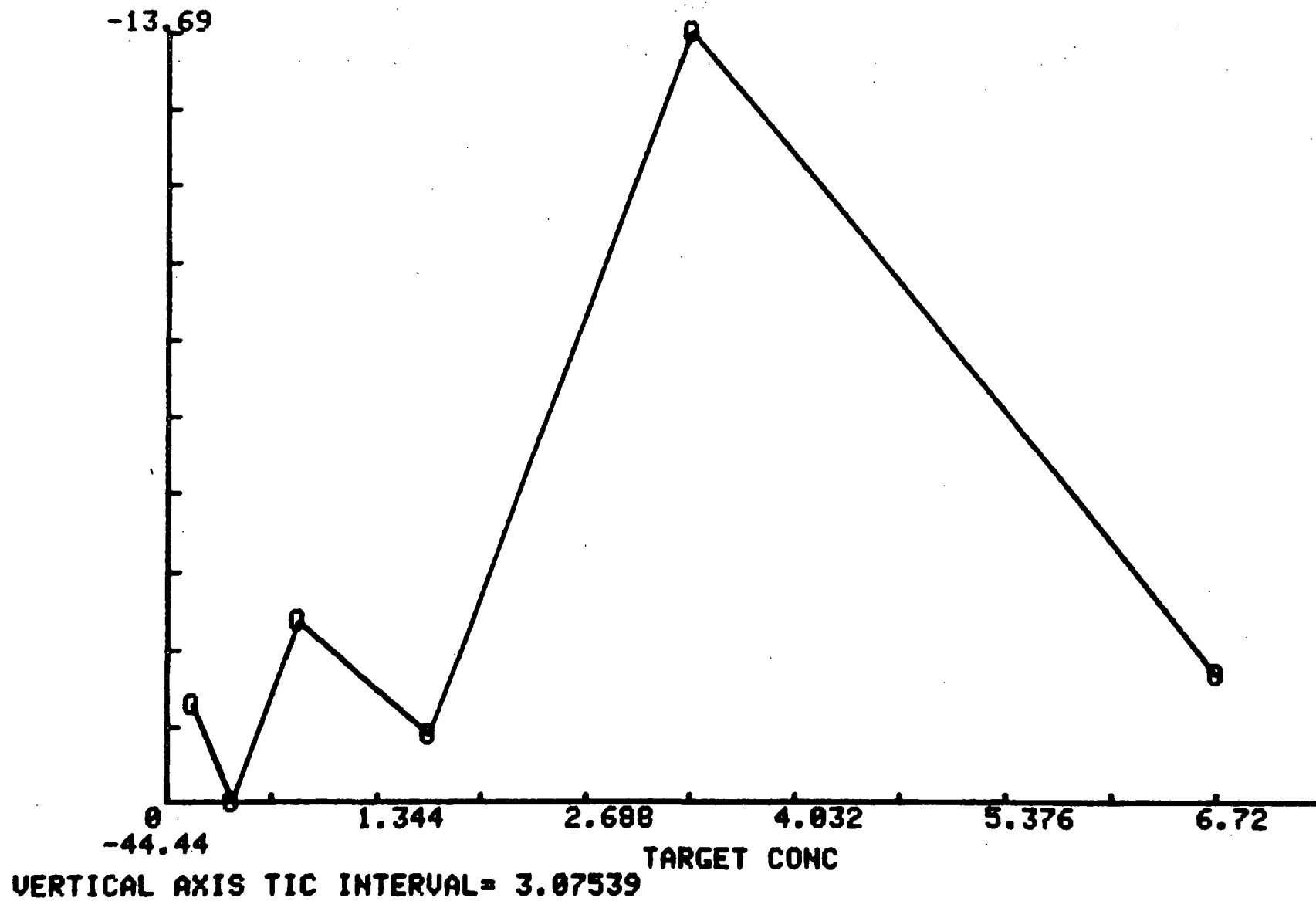
VERTICAL AXIS TIC INTERVAL = 0.437

TNT IN WATER  
RUNS 1 2 3  
STANDARD DEVIATION

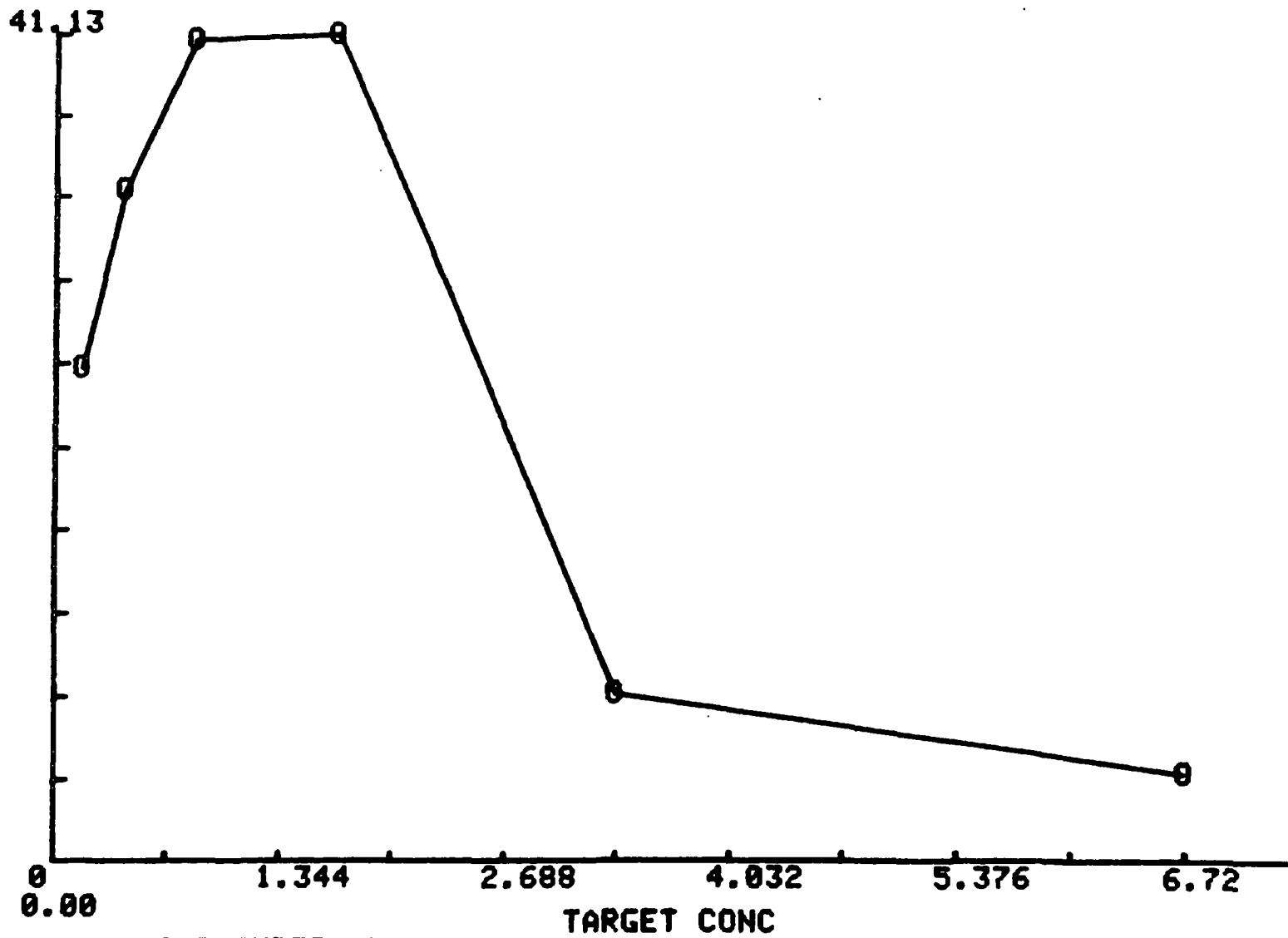


VERTICAL AXIS TIC INTERVAL= 0.0403

TNT IN WATER  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



TNT IN WATER  
RUNS 1 2 3  
PERCENT IMPRECISION



VERTICAL AXIS TIC INTERVAL = 4.11276

TETRYL IN WATER

1

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/L	Day 1 Found Conc. UG/L	Day 2 Found Conc. UG/L	Day 3 Found Conc. UG/L	Day 4 Found Conc. UG/L
0.20	0.12	0.16	0.15	
0.51	0.40	0.40	0.30	
1.02	1.10	0.80	0.93	
2.04	1.75	1.33	1.86	
4.08	4.00	4.20	4.00	
8.16	6.85	8.54	7.96	

TETRYL IN WATER

1

TARGET CONC

ANALYSIS OF 18 TARGET CONC-FOUND CONC POINTS

MEAN= 2.669 SD= 2.84897349935

FOUND CONC

MEAN= 2.49166666667 SD= 2.79266739453

NO. RUNS 3 TOTAL X-Y ALL RUNS 18 NO. CONCENTR 18  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.104550047125

SLOPE= 0.972730128809

R= 0.99234243377

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.12642184213

COMPUTE T

D.F.= 16

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1

t= 1.74588066418

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.548633296764

x(d)= 1.33116414808



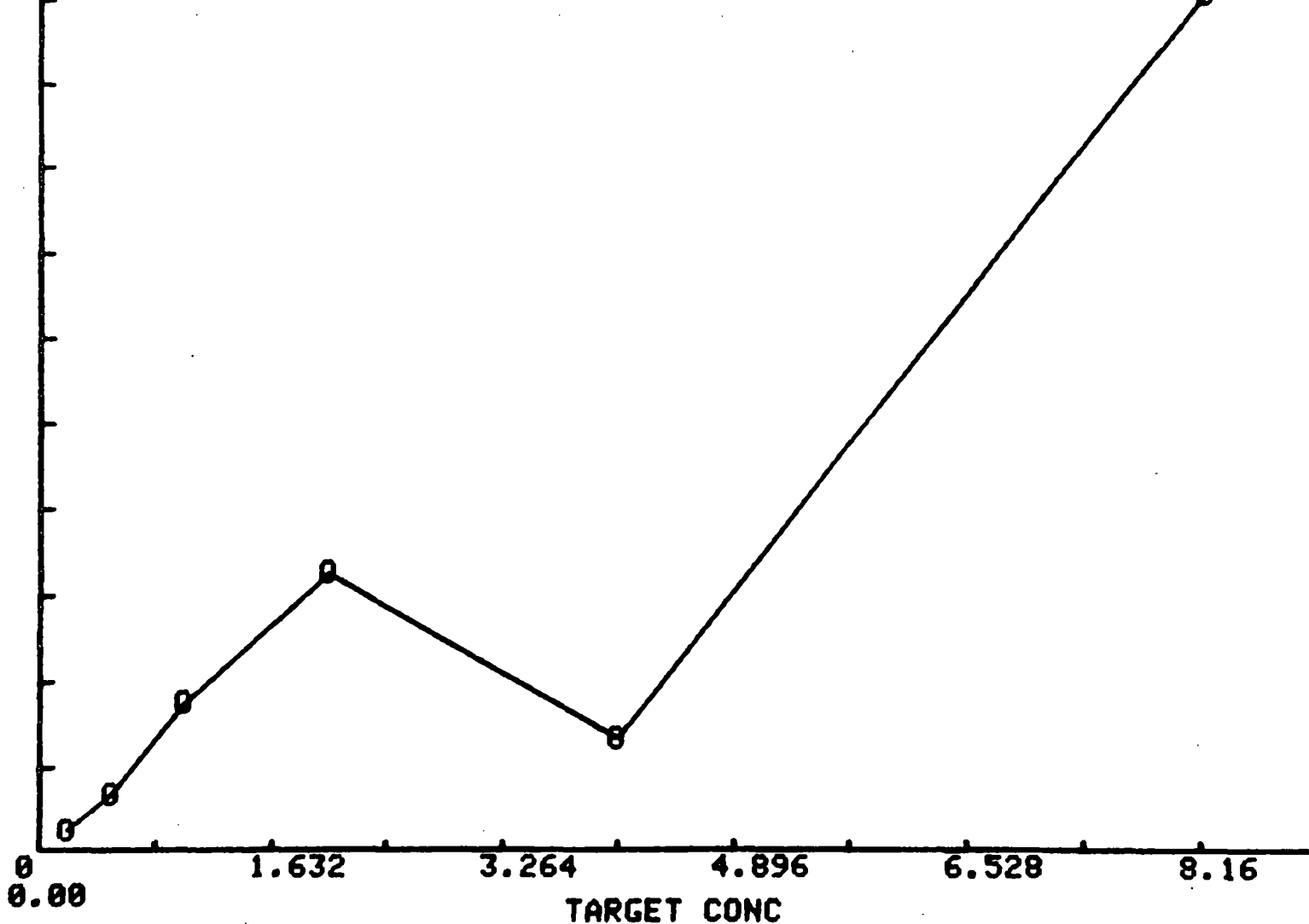
# TETRYL IN WATER

## 1 STATISTICAL DATA USED TO DETERMINE PERCENT INACCURACY AND COEFFICIENT OF VARIATION

Target Conc. UG/L	Mean Found Conc. UG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.20	0.14	0.01	-29.74	10.27
0.51	0.37	0.04	-28.10	11.13
1.02	0.94	0.11	-7.52	11.28
2.04	1.65	0.20	-19.28	12.01
4.08	4.07	0.08	-0.33	2.01
8.16	7.78	0.61	-4.62	7.80

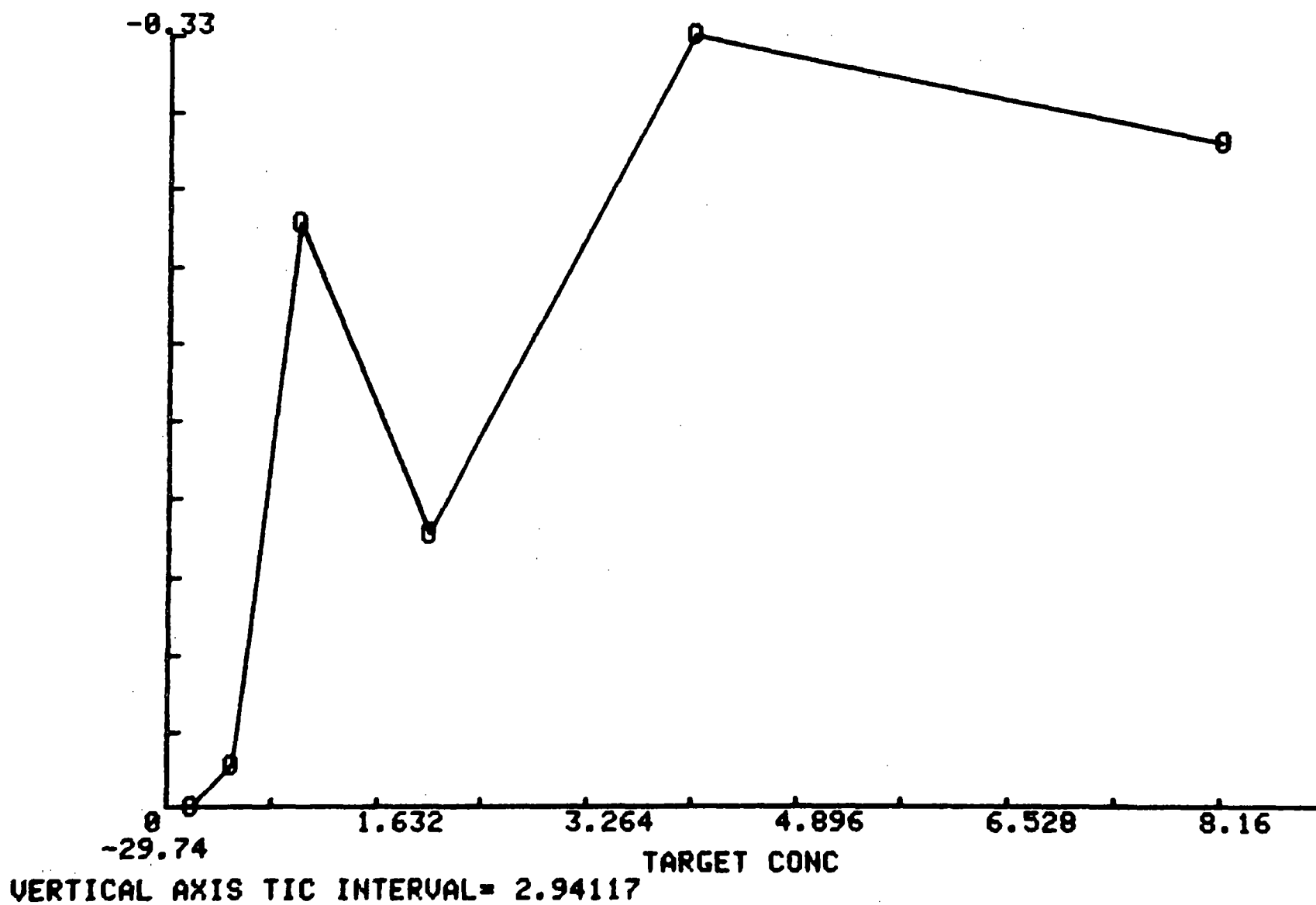
TETRYL IN WATER  
1  
STANDARD DEVIATION

0.61

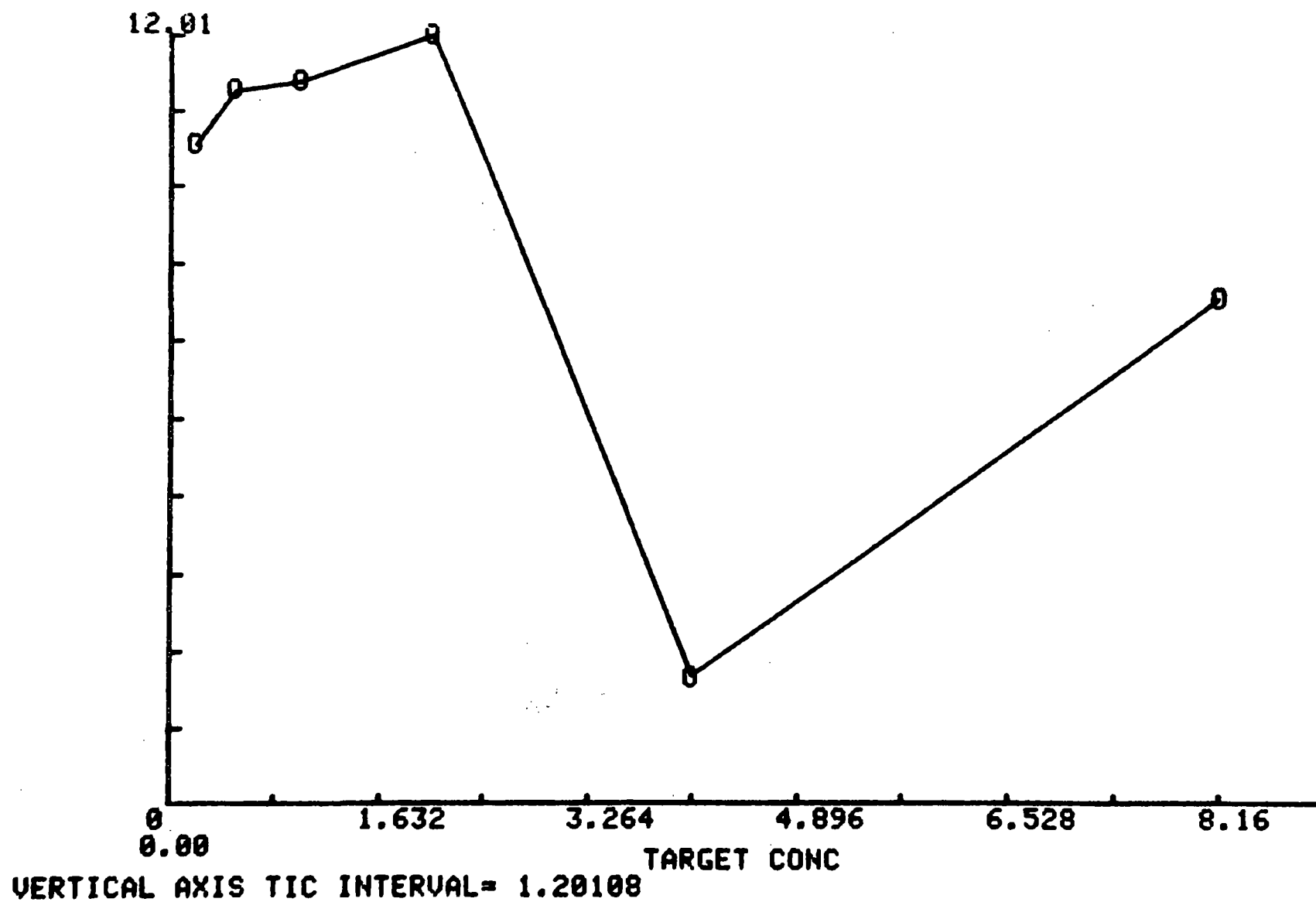


VERTICAL AXIS TIC INTERVAL = 0.06072

TETRYL IN WATER  
1  
MEAN PERCENT INACCURACY



TETRYL IN WATER  
1  
PERCENT IMPRECISION



2,4-DNT IN SOIL

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/G	Day 1 Found Conc. UG/G	Day 2 Found Conc. UG/G	Day 3 Found Conc. UG/G	Day 4 Found Conc. UG/G
0.51	0.50	0.50	0.50	
1.02	0.98	1.10	1.10	
2.55	2.50	2.50	2.70	
5.10	4.90	4.80	5.00	

2,4-DNT IN SOIL

RUNS 1 2 3

TARGET CONC

ANALYSIS OF 12 TARGET CONC-FOUND CONC POINTS

MEAN= 2.295 SD= 1.86437169529

FOUND CONC

MEAN= 2.25666666667 SD= 1.78019066054

N0. RUNS 3 TOTAL X-Y ALL RUNS 12 N0. CONCENTR 12

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0678911564626

SLOPE= 0.953714819261

R= 0.998813752828

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00826557823152

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

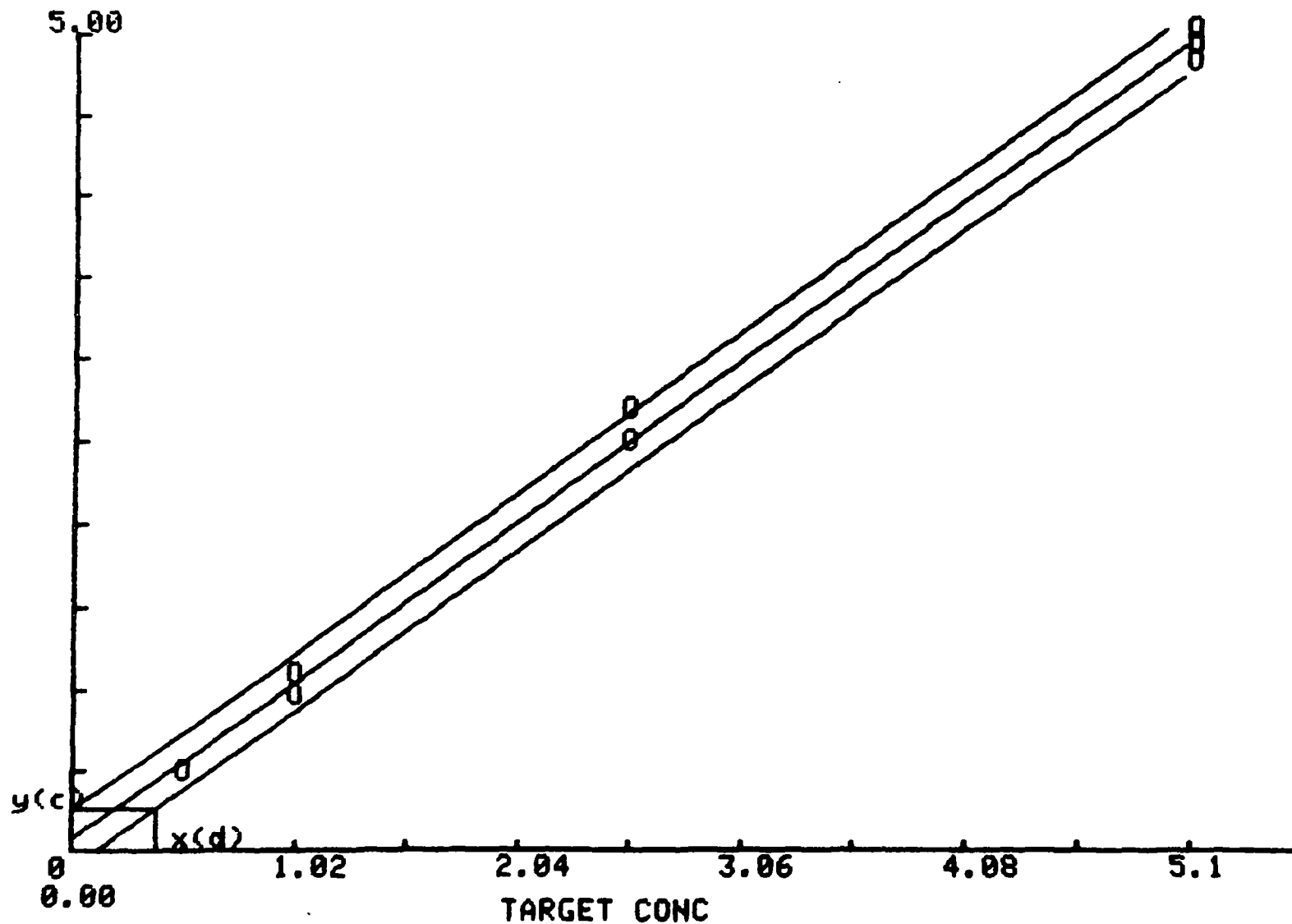
t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.249977885915

x(d)= 0.378559437535

2,4-DNT IN SOIL  
RUNS 1 2 3  
FOUND CONC



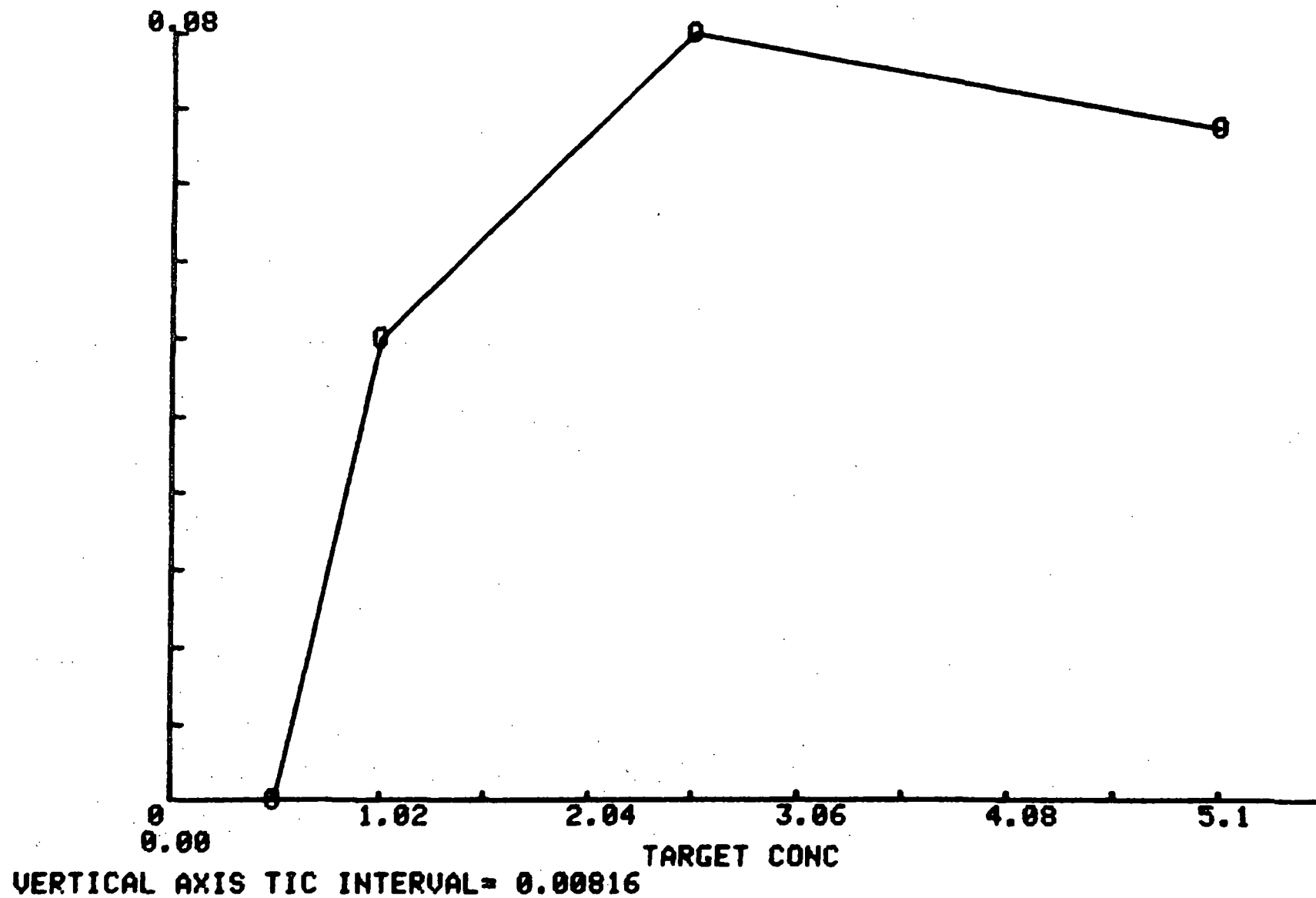
**2,4-DNT IN SOIL**

**RUNS 1 2 3**

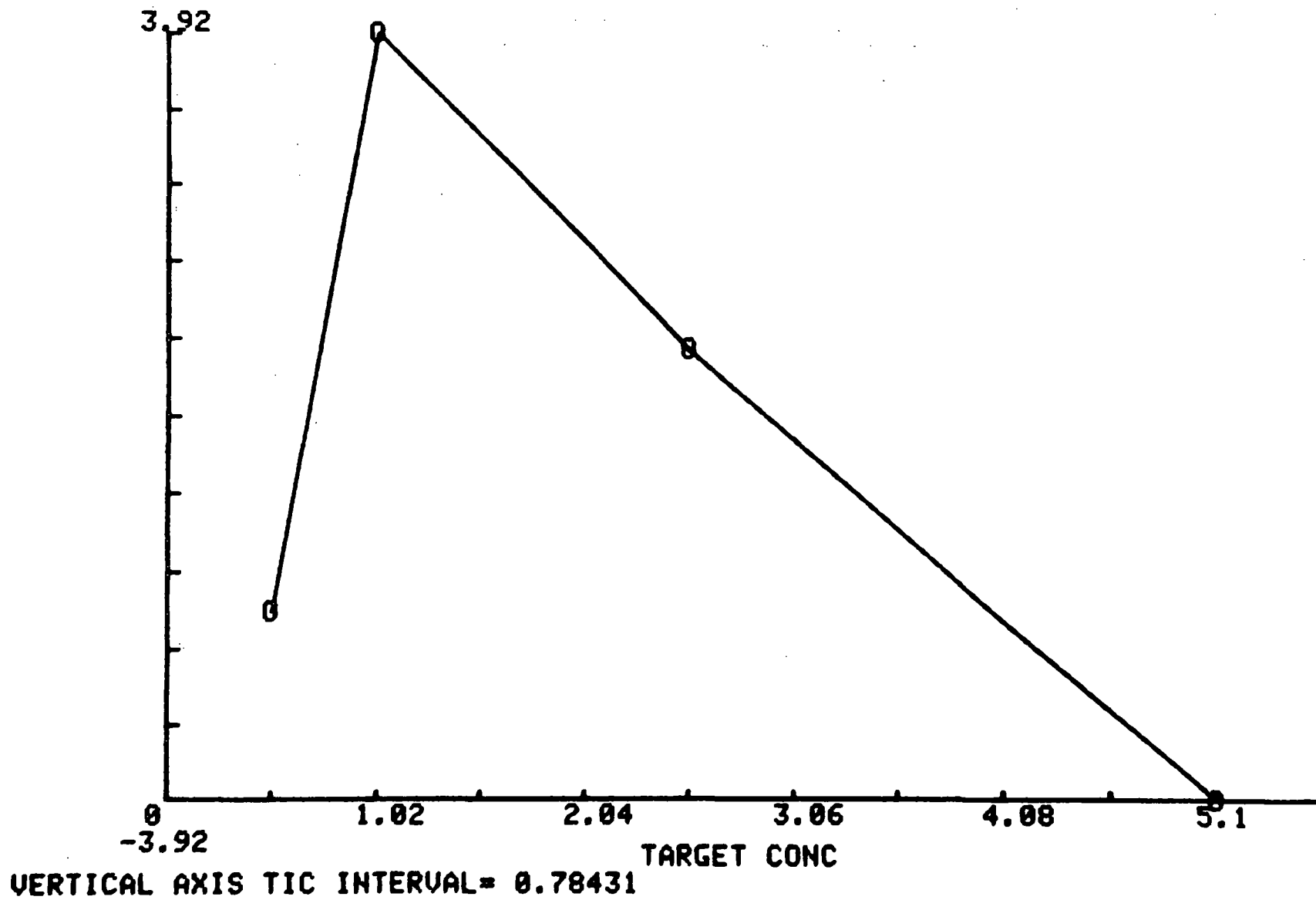
**STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION**

<b>Target Conc. UG/G</b>	<b>Mean Found Conc. UG/G</b>	<b>Standard Deviation</b>	<b>Percent Inaccuracy</b>	<b>Coefficient(% of variation</b>
<b>0.51</b>	<b>0.50</b>	<b>0.00</b>	<b>-1.96</b>	<b>0.00</b>
<b>1.02</b>	<b>1.06</b>	<b>0.05</b>	<b>3.92</b>	<b>4.62</b>
<b>2.55</b>	<b>2.57</b>	<b>0.08</b>	<b>0.65</b>	<b>3.18</b>
<b>5.10</b>	<b>4.90</b>	<b>0.07</b>	<b>-3.92</b>	<b>1.44</b>

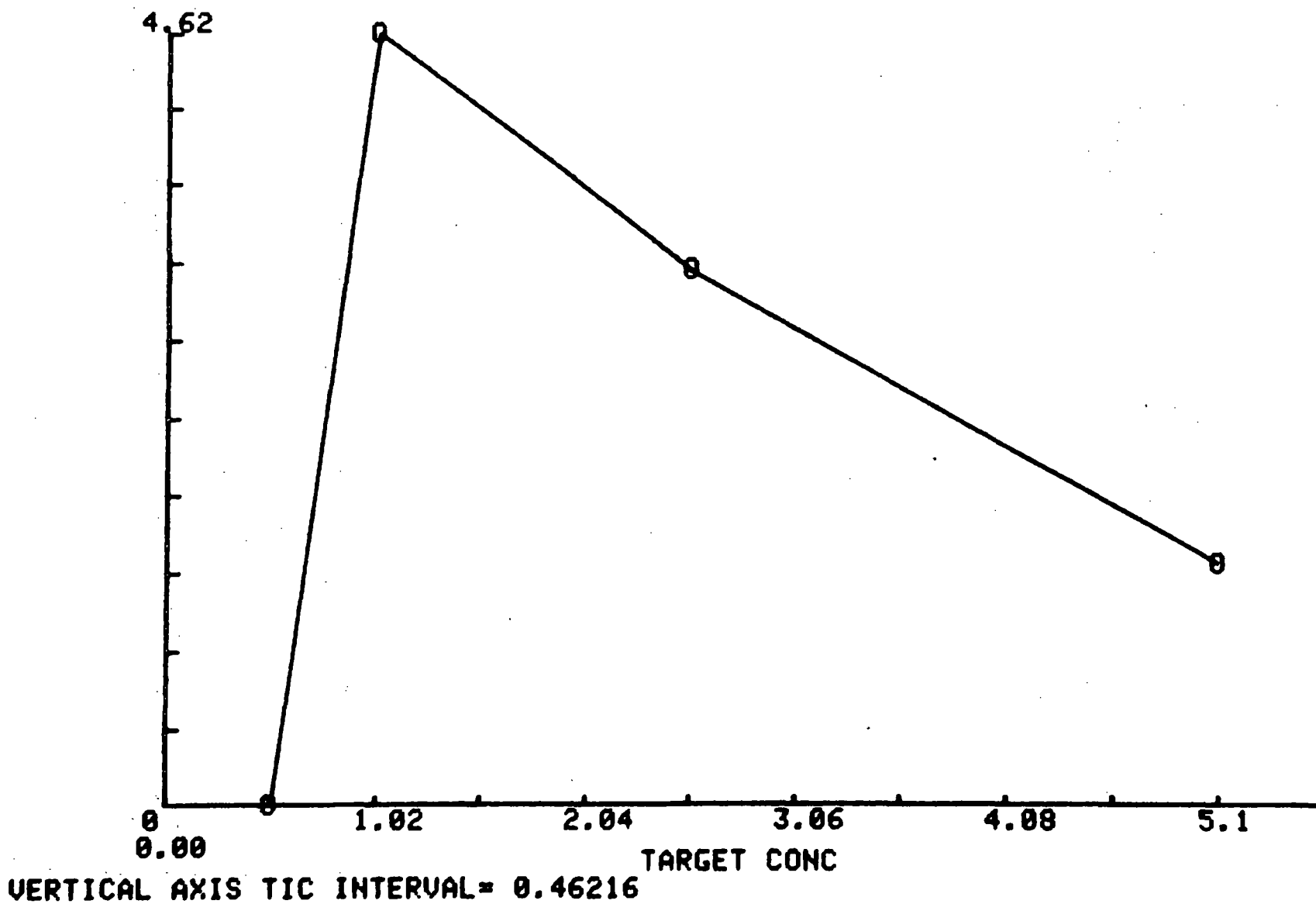
2,4-DNT IN SOIL  
RUNS 1 2 3  
STANDARD DEVIATION



2,4-DNT IN SOIL  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



2,4-DNT IN SOIL  
RUNS 1 2 3  
PERCENT IMPRECISION



TNT IN SOIL

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/G	Day 1 Found Conc. UG/G	Day 2 Found Conc. UG/G	Day 3 Found Conc. UG/G	Day 4 Found Conc. UG/G
0.42	0.30	0.35	0.32	
0.84	0.58	0.75	0.75	
2.10	1.00	1.90	2.00	
4.20	2.80	4.10	4.20	

TNT IN SOIL  
RUNS 1 2 3  
TARGET CONC  
ANALYSIS OF 12 TARGET CONC-FOUND CONC POINTS

MEAN= 1.89 SD= 1.53536492553

FOUND CONC  
MEAN= 1.5875 SD= 1.42810825284

NO. RUNS 3 TOTAL X-Y ALL RUNS 12 NO. CONCENTR 12  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.0932653061225  
SLOPE= 0.889293812763  
R= 0.956083354252  
MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.19272207483

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1

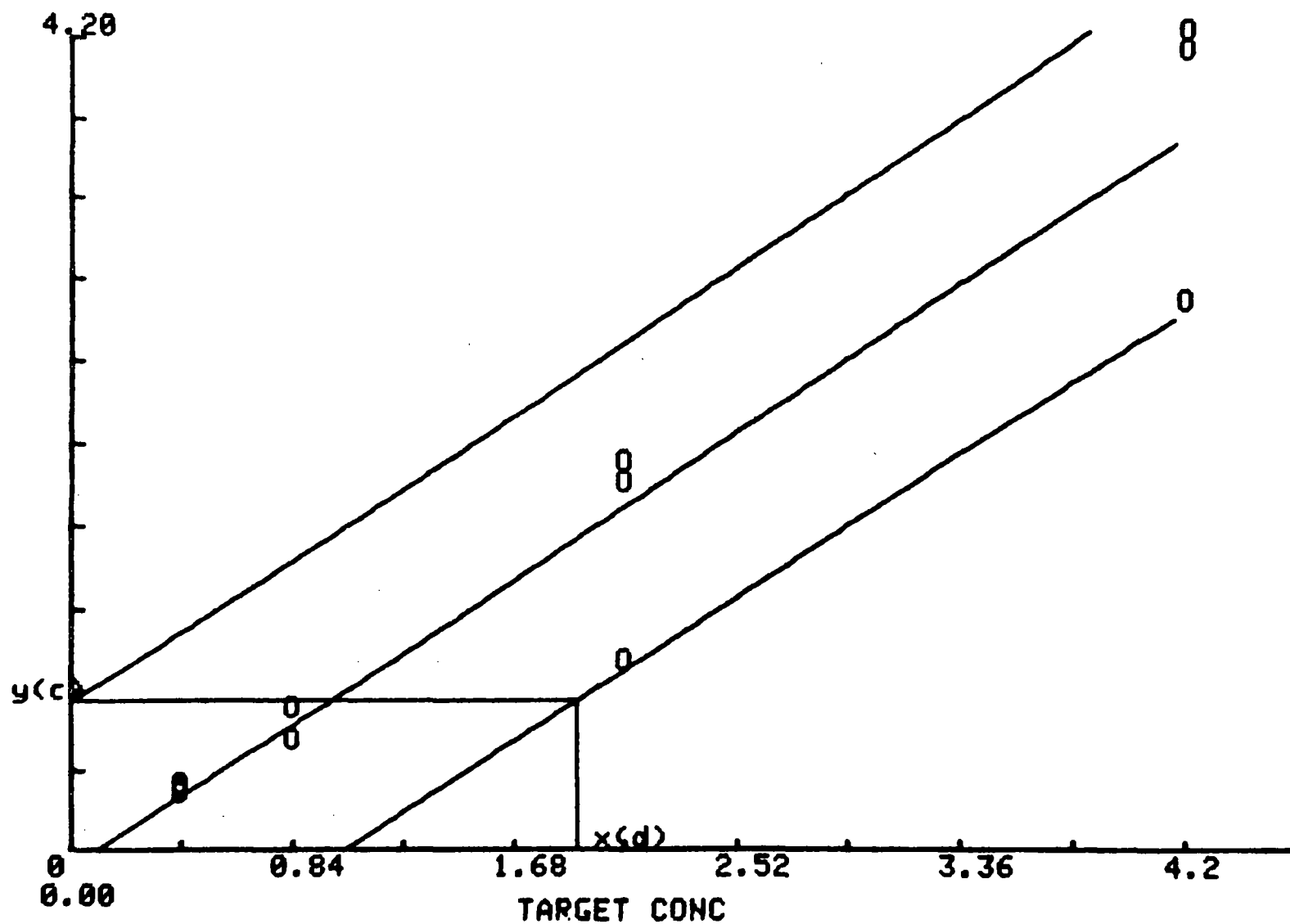
t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.785974630623

x(d)= 1.92018986336

TNT IN SOIL  
RUNS 1 2 3  
FOUND CONC



VERTICAL AXIS TIC INTERVAL= 0.42

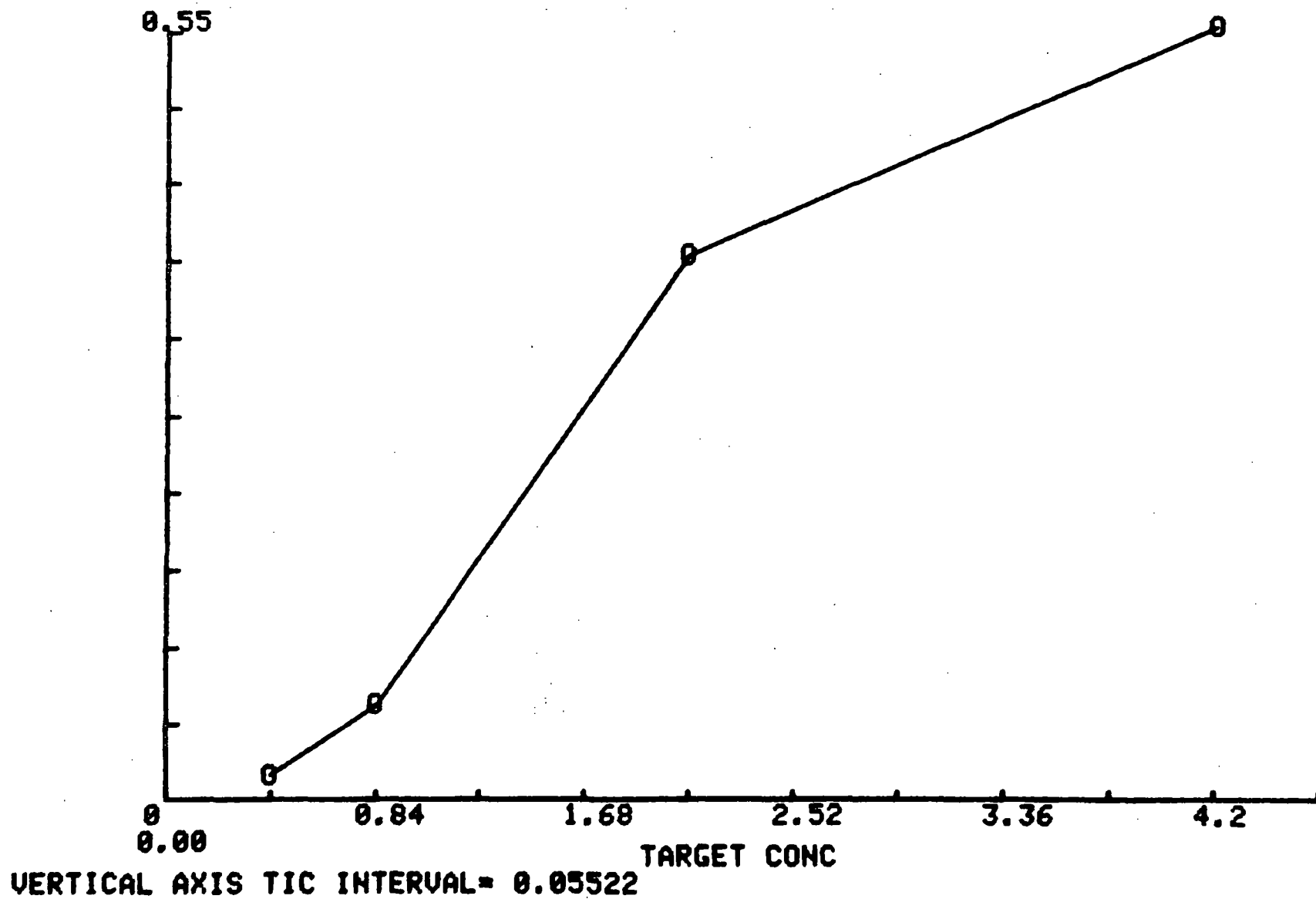
**TNT IN SOIL**

**RUNS 1 2 3**

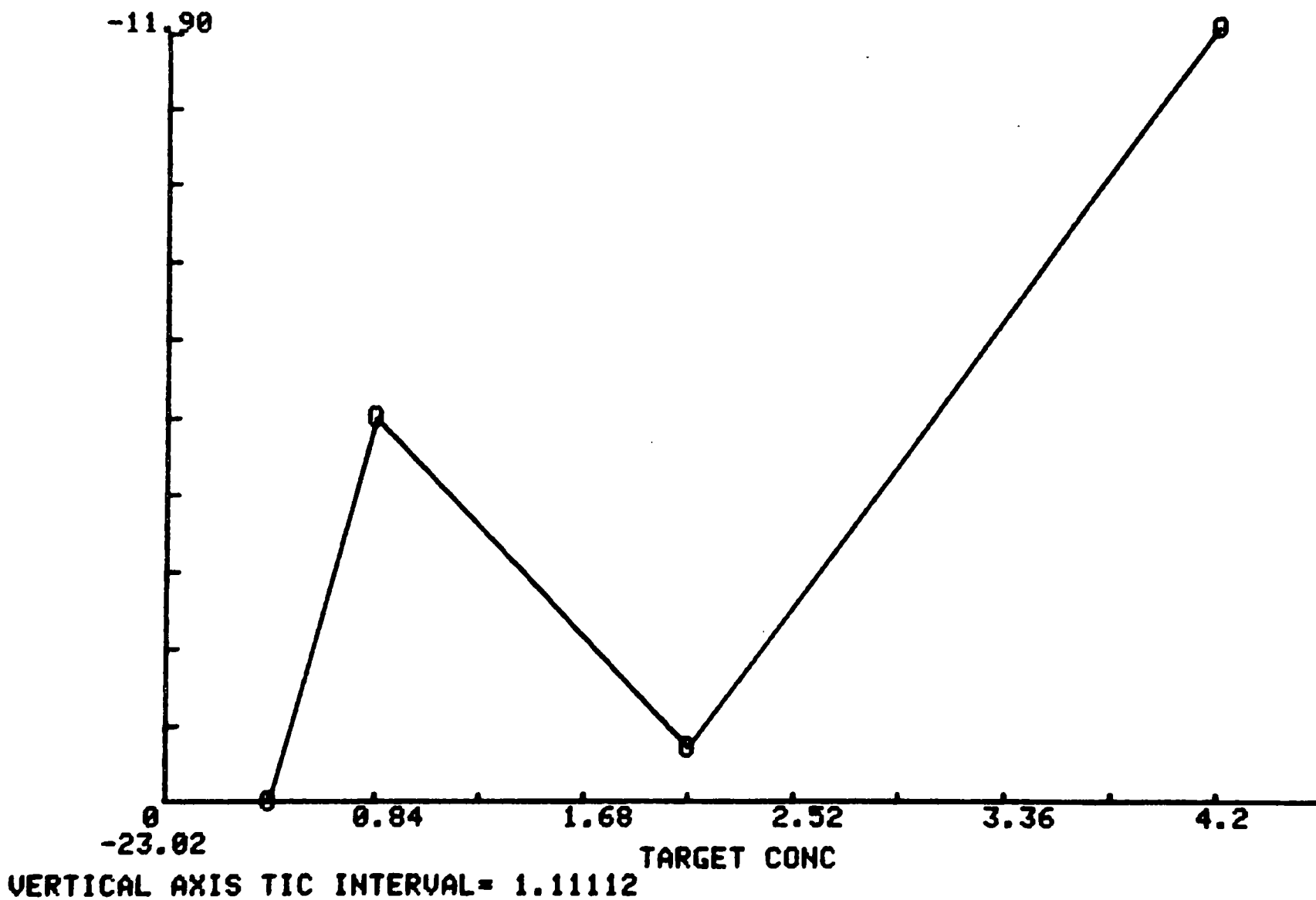
**STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION**

<b>Target Conc. UG/G</b>	<b>Mean Found Conc. UG/G</b>	<b>Standard Deviation</b>	<b>Percent Inaccuracy</b>	<b>Coefficient(%) of variation</b>
<b>0.42</b>	<b>0.32</b>	<b>0.02</b>	<b>-23.02</b>	<b>5.50</b>
<b>0.84</b>	<b>0.69</b>	<b>0.07</b>	<b>-17.46</b>	<b>10.01</b>
<b>2.10</b>	<b>1.63</b>	<b>0.39</b>	<b>-22.22</b>	<b>23.84</b>
<b>4.20</b>	<b>3.70</b>	<b>0.55</b>	<b>-11.90</b>	<b>14.93</b>

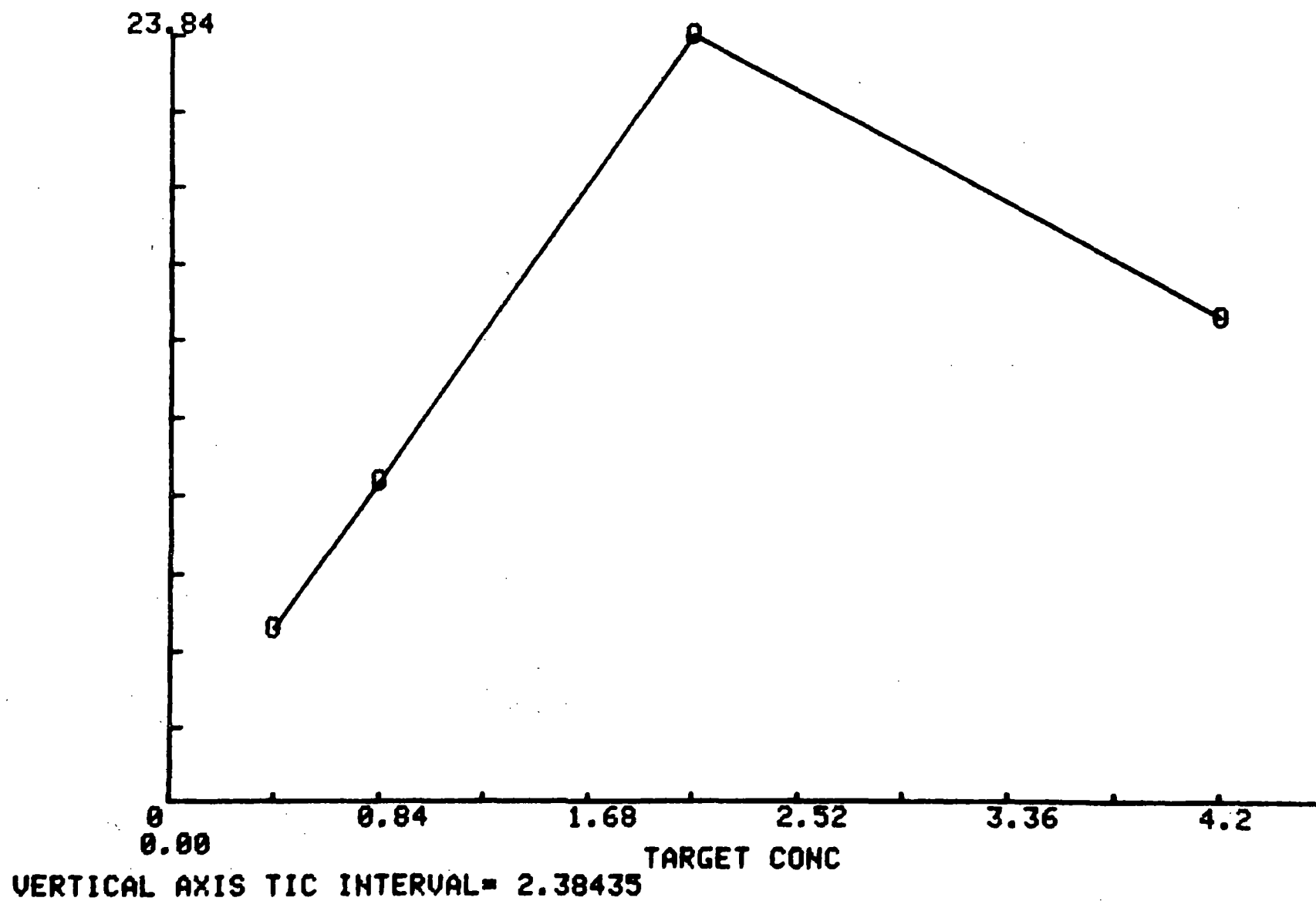
TNT IN SOIL  
RUNS 1 2 3  
STANDARD DEVIATION



TNT IN SOIL  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



TNT IN SOIL  
RUNS 1 2 3  
PERCENT IMPRECISION



TETRYL IN SOIL

RUNS 1 2 3

COMPILATION OF TARGET CONC. VS FOUND CONC

Target Conc. UG/G	Day 1 Found Conc. UG/G	Day 2 Found Conc. UG/G	Day 3 Found Conc. UG/G	Day 4 Found Conc. UG/G
0.51	0.25	0.30	0.30	
1.02	0.50	0.65	0.75	
2.55	1.60	1.60	1.90	
5.10	4.80	4.50	4.90	

TETRYL IN SOIL

RUNS 1 2 3

TARGET CONC

ANALYSIS OF 12 TARGET CONC-FOUND CONC POINTS

MEAN= 2.295 SD= 1.86437169529

FOUND CONC

MEAN= 1.8375 SD= 1.83378954379

NØ. RUNS 3 TOTAL X-Y ALL RUNS 12 NØ. CONCENTR 12  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.394897959184

SLOPE= 0.972722422302

R= 0.988944537094

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.0813375850342

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

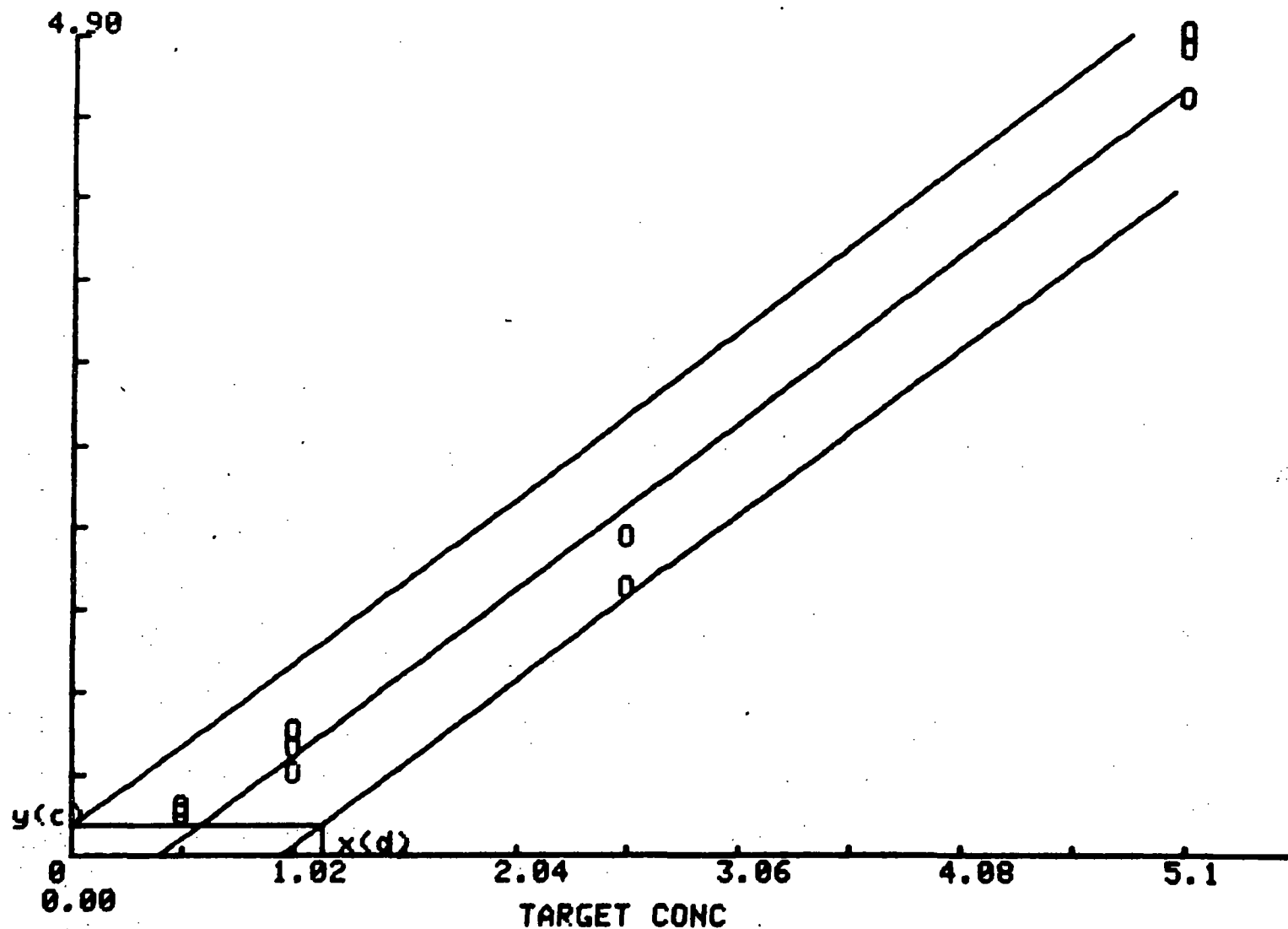
t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.176300874419

x(d)= 1.14902500285

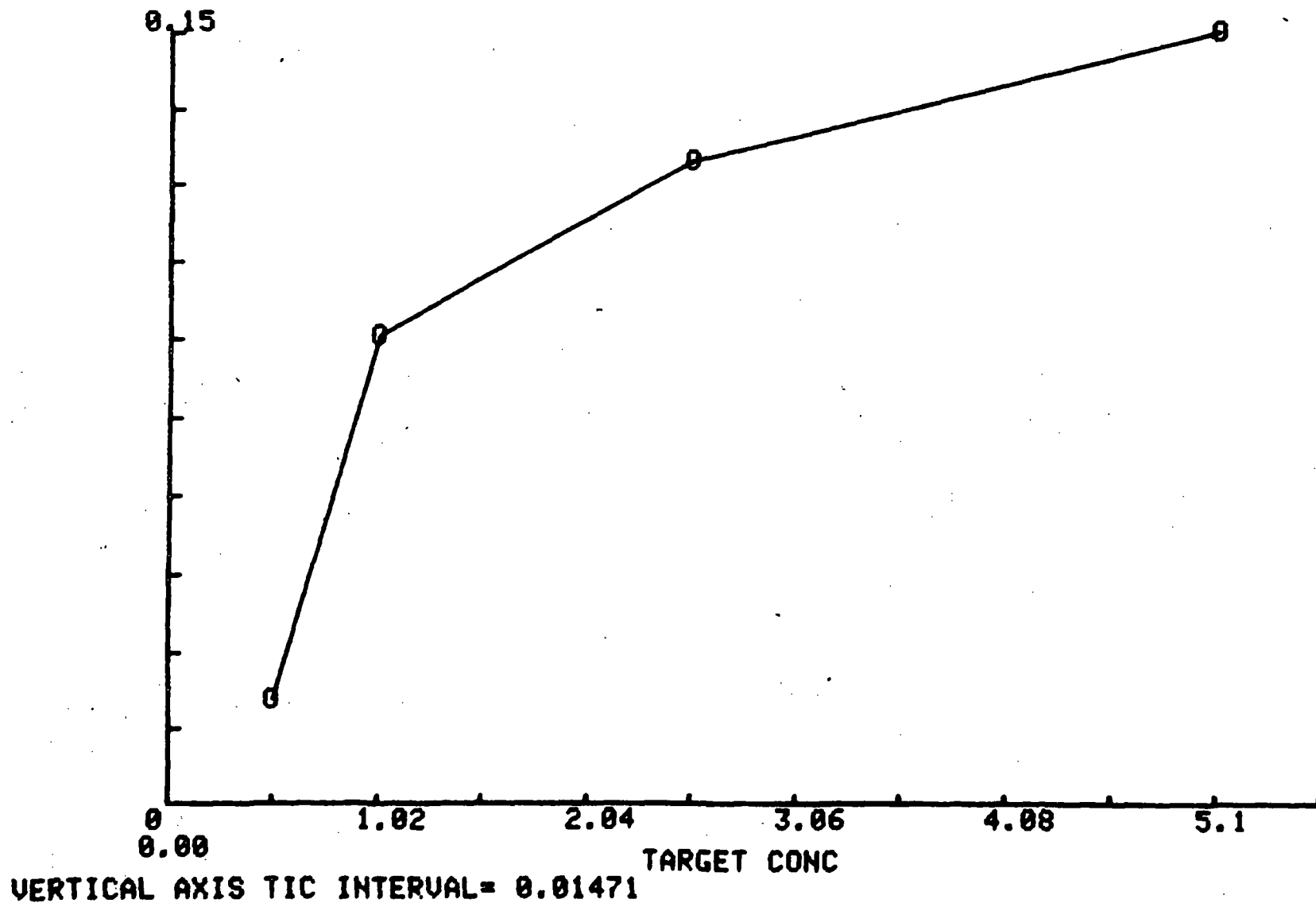
TETRYL IN SOIL  
 RUNS 1 2 3  
 FOUND CONC



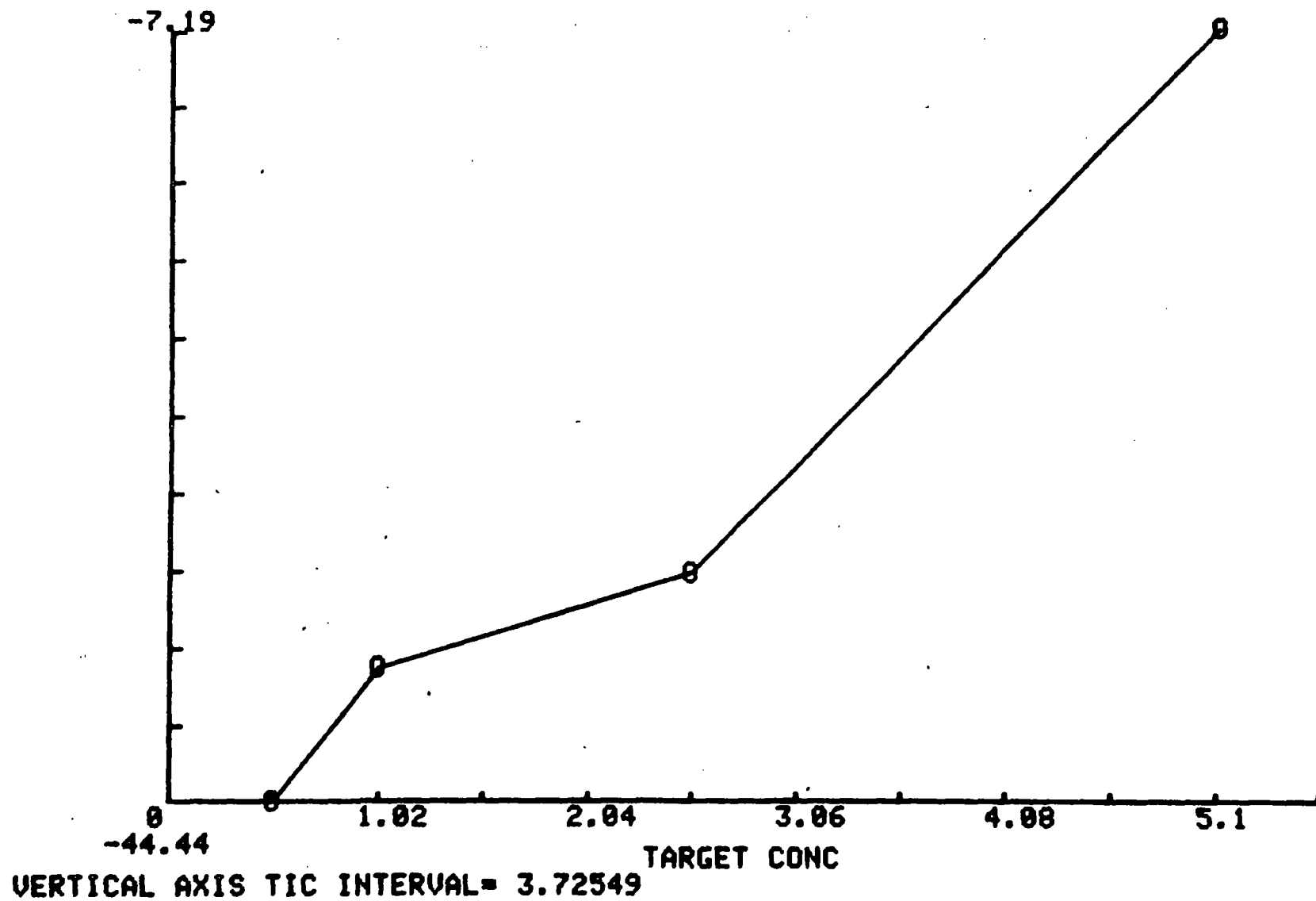
**TETRYL IN SOIL****RUNS 1 2 3****STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION**

<b>Target Conc. UG/G</b>	<b>Mean Found Conc. UG/G</b>	<b>Standard Deviation</b>	<b>Percent Inaccuracy</b>	<b>Coefficient(%) of variation</b>
0.51	0.28	0.02	-44.44	7.20
1.02	0.63	0.09	-37.91	14.05
2.55	1.70	0.12	-33.33	7.20
5.10	4.73	0.15	-7.19	3.11

TETRYL IN SOIL  
RUNS 1 2 3  
STANDARD DEVIATION

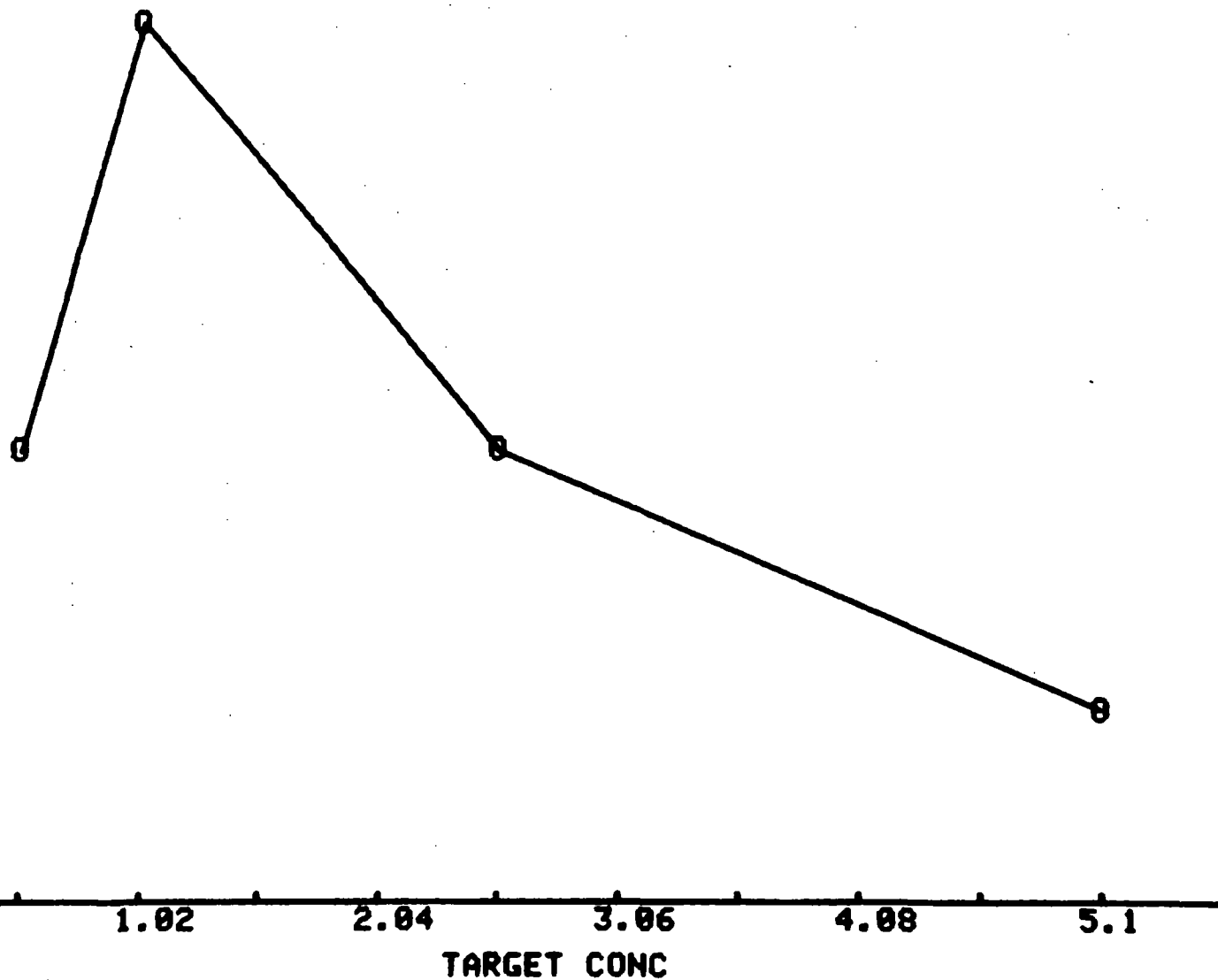


TETRYL IN SOIL  
RUNS 1 2 3  
MEAN PERCENT INACCURACY



TETRYL IN SOIL  
RUNS 1 2 3  
PERCENT IMPRECISION

14.05



VERTICAL AXIS TIC INTERVAL = 1.40487

DETERMINATION OF NITROCELLULOSE, NITROGLYCERINE  
AND PETN IN WATER

I. Application: This method is used to qualitatively determine the concentration of nitrocellulose (NC), nitroglycerine (NG) and PETN in water samples.

A. Tested Concentration Range - Response is linear from 10 to 50 mg/liter in water.

B. Sensitivity Limits - Response for 5 µg of compound is: NC = 0.089, NG = 0.17, PETN = 0.15 absorbance units.

C. Detection Limits -

<u>Compound</u>	<u>Water (µg/l)</u>
NC	9,300
NG	4,700
PETN	2,900

D. Interferences - Nitrite ion will interfere.

E. Analysis Rate - Approximately 20 samples can be analyzed by one worker in an eight hour day.

II. Chemistry

Nitrite ion is cleaved from the nitrate ester in basic solution which diazotizes procaine, in acidic solution, which in turn couples with N,N-dimethyl-1-naphthylamine to produce an azo-dye. This dye is determined from its absorbance at 510 µm.

$C_3H_5N_3O_9$  - Trinitroglycerine; glyceryl trinitrate; CAS RN-55-63-0  
mp = 13°C, decomposition pt = 145°C;

$C_5H_8N_4O_{12}$  - Pentaerythritol tetranitrate CAS RN-78-11-5  
mp = 140°C; explosive decomposition pt. 210°C;

$(C_6H_{12}N_5O_{16})_x$  - Nitrocellulose, soluble gun cotton, CAS RN-9004-70-0  
mp = decomposes on heating.

Handling Hazards: Explosive hazard, avoid heat, shock or open flame. Toxic inhalation and skin absorption hazards exist. Do not handle NG except as a dilute solution.

III. Apparatus

A. Instrumentation - Absorbance measurements are made at 510 nm on a Bausch and Lomb Spectronic 20 spectrometer.

B. Parameters - N/A

C. Hardware/Glassware.

1. 0.1, 1, and 2 ml pipets
2. 13 x 100 mm glass test tubes
3. 10 ml volumetric flasks
4. 100 ml volumetric flasks
5. 1 cm spectrometer cell (glass)
6. water bath
7. hot plate
8. 25  $\mu$ l graduated syringe
9. 8 ounce glass bottle
10. 2 dram glass vials

D. Chemicals

1. KOH, Analytical reagent grade
2. Glacial acetic acid, ACS grade
3. Acetone, ACS grade
4. N,N-dimethyl-1-naphthylamine
5. Procaine

E. Reagents

1. 10 percent KOH: Weigh 10.0 g of reagent grade potassium hydroxide into a 100 ml volumetric flask and dilute slowly with deionized water (nitrite free).
2. 20 percent KOH: Same as above except use 20.0 g of KOH.
3. 10 percent Acetic Acid: Pipet 10.0 ml of glacial acetic acid into a 100 ml volumetric flask which is partially filled with deionized water (nitrite free). Dilute to volume with deionized water.
4. 50 percent Acetic Acid: Same as above except use 50 ml of glacial acetic acid.
5. Color Developing Reagent. Weigh 0.35 g of each of procaine and N,N-dimethyl-1-naphthylamine into a 100 ml volumetric flask and dilute to volume with 50 percent acetic acid-water.
6. Working Solution of Color Reagent. Pipette 20 ml of the color developing reagent into a 100 ml volumetric flask. Dilute to volume with deionized water (nitrite free).

IV. Standards

A. Calibration Standards

1. Stock - Weigh 10.0 mg of NC, PETN, or 1 ml of a 1% NG in acetone SARM into a 10.0 ml volumetric flask and dilute to volume with acetone to obtain a 1  $\mu$ g/ $\mu$ l solution. This solution is refrigerated when not in use.

2. Working - Prepare standard curve daily by adding 0.0, 5.0, 10.0 and 25.0  $\mu$ l of 1  $\mu$ g/ $\mu$ l stock solution in duplicate into 13 mm by 100 mm test tubes, using a 25  $\mu$ l syringe. Add 0.5 ml of deionized water to each tube and mix thoroughly.

- B. Control Spikes - For water samples spike as indicated for the working standards.

## V. Procedure

- A. Water Sample Handling/Preparation - Refrigerate the samples until ready for analysis. Prior to analysis, filter through a 0.45 micron Millipore filter and store refrigerated in a 2 dram glass vial.

- B. Analysis of Samples

1. Water - Pipet 0.50 ml of water into a 13 x 100 mm test tube and add 0.50 ml of 20 percent potassium hydroxide solution. Go to step 2.
2. Mix sample well and place in boiling water bath for 30 minutes.
3. Cool the sample, add 2.0 ml of 10 percent acetic acid, and mix.
4. Add 1.0 ml of color developing reagent working solution and mix well.
5. Allow color to develop for 1.5 hour, transfer the solution to a clean, dry spectrophotometer cell and read the absorbance at 510 nm.

## VI. Calculations

Construct a calibration curve of absorbance vs. quantity ( $\mu$ g) of the compound of interest (differing responses will be observed for NC,

PETN, and NG). Determine concentration of compound in samples by interpolating from the calibration curve.

For water: Concentration ( $\mu\text{g/l}$ ) =  $\mu\text{g}$  (from graph) X 2000

For soil: Concentration ( $\mu\text{g/l}$ ) =  $\mu\text{g}$  (from graph) X 100.

VII. REFERENCE None.

# NITROCELLULOSE IN WATER

RUNS 1 2

## COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. MG/L	Day 1 Found Conc. MG/L	Day 2 Found Conc. MG/L	Day 3 Found Conc. MG/L	Day 4 Found Conc. MG/L
0.00	1.64	2.43		
5.00	6.47	6.37		
10.00	11.31	9.52		
20.00	16.46	17.72		
50.00	47.25	47.05		
100.00	101.88	101.91		

NITROCELLULOSE IN WATER

RUNS 1 2

TARGET CONC

ANALYSIS OF 12 TARGET CONC-PEAK AREA POINTS

MEAN= 30.8333333333 SD= 36.4837032942

PEAK AREA

MEAN= 0.193166666667 SD= 0.229348021905

NO. RUNS 2 TOTAL X-Y ALL RUNS 12 NO. CONCENTR 12

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -2.743312464E-4

SLOPE= 0.00627376209448

R= 0.998003265486

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 2.308336938E-4

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

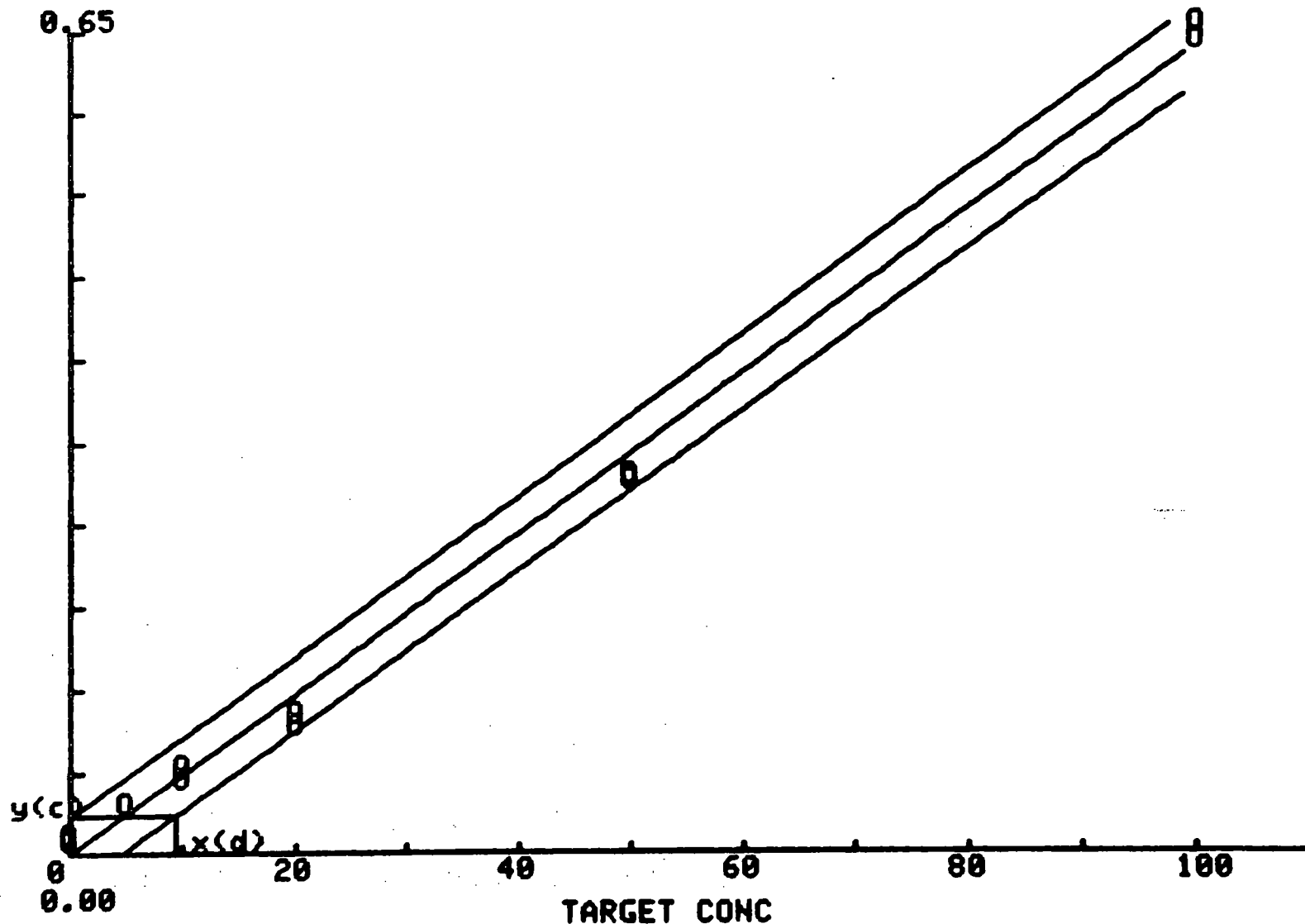
y(c)= 0.0292335894436

x(d)= 9.33791924766

# NITROCELLULOSE IN WATER

RUNS 1 2

PEAK AREA



NITROGLYCERIN IN WATER

RUNS 1 2

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. MG/L	Day 1 Found Conc. MG/L	Day 2 Found Conc. MG/L	Day 3 Found Conc. MG/L	Day 4 Found Conc. MG/L
0.00	-1.23	0.08		
5.00	5.35	5.27		
10.00	11.93	9.42		
20.00	18.95	20.22		

NITROGLYCERIN IN WATER

RUNS 1 2

TARGET CONC

ANALYSIS OF 8 TARGET CONC-PEAK AREA POINTS

MEAN= 8.75 SD= 7.90569415042

PEAK AREA

MEAN= 0.056125 SD= 0.0373647561212

NO. RUNS 2 TOTAL X-Y ALL RUNS 8 NO. CONCENTR 8

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0151

SLOPE= 0.00468857142857

R= 0.992015352553

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 2.590714286E-5

COMPUTE T

D.F.= 6

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

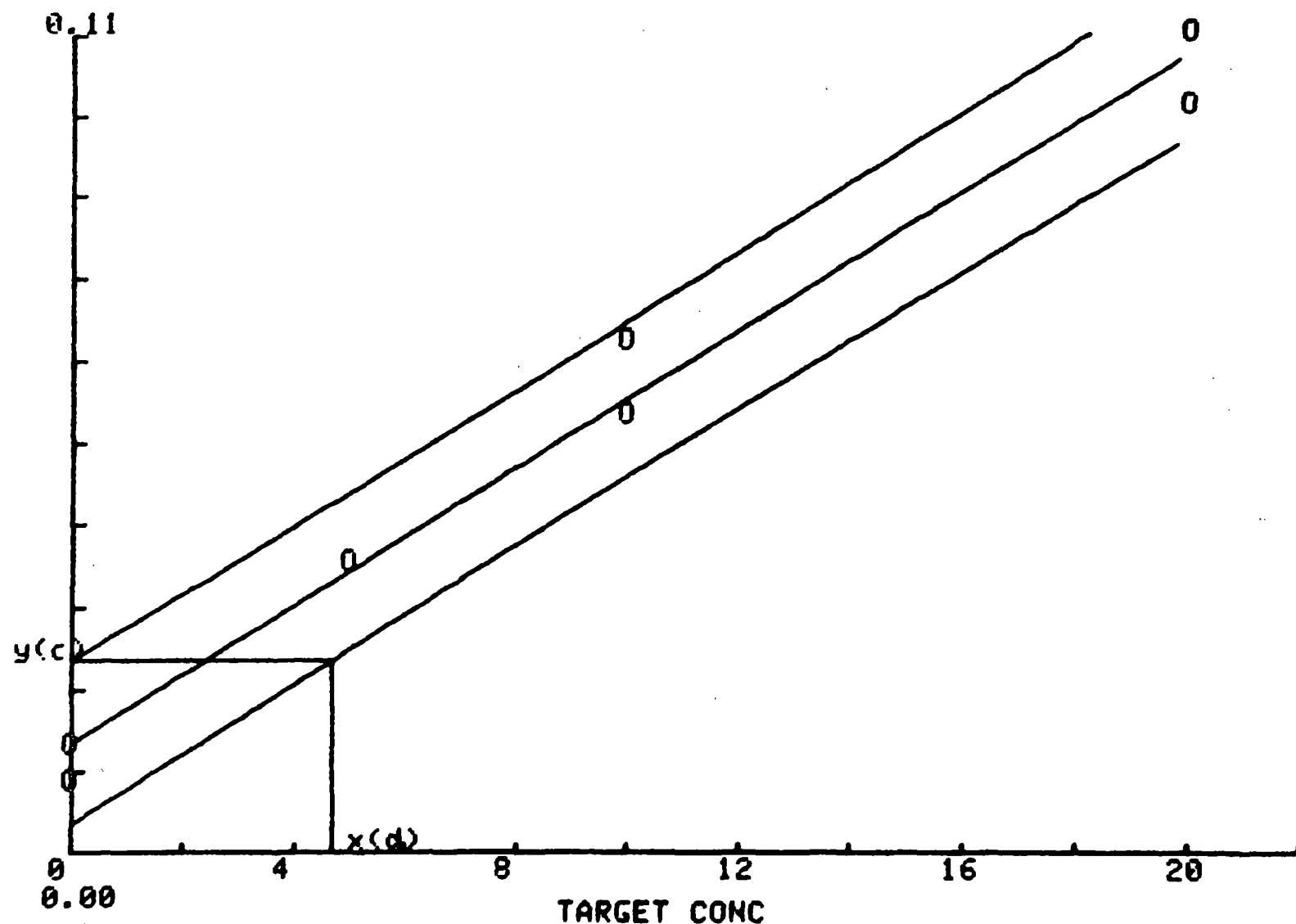
t= 1.94317596321

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.0263769996195

x(d)= 4.68006454046

NITROGLYCERIN IN WATER  
RUNS 1 2  
PEAK AREA



VERTICAL AXIS TIC INTERVAL = 0.0112

NITROGLYCERIN IN WATER

RUNS 1 2

COMPILATION OF TARGET CONC. VS ST PEAK AREA

Target Conc. MG/L	Day 1 Found Conc. MG/L	Day 2 Found Conc. MG/L	Day 3 Found Conc. MG/L	Day 4 Found Conc. MG/L
0.00	0.44	1.12		
5.00	2.11	2.58		
10.00	6.10	5.92		
20.00	19.26	17.85		
50.00	62.82	63.13		
100.00	94.27	94.39		

NITROGLYCERIN IN WATER

RUNS 1 2

TARGET CONC

ANALYSIS OF 12 TARGET CONC-ST PEAK AREA POINTS

MEAN= 30.8333333333 SD= 36.4837032942

ST PEAK AREA

MEAN= 0.585 SD= 0.708824763573

N0. RUNS 2 TOTAL X-Y ALL RUNS 12 N0. CONCENTR 12

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.00495731360273

SLOPE= 0.0191337507114

R= 0.984827449231

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.016643773819

COMPUTE T

D.F.= 10

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

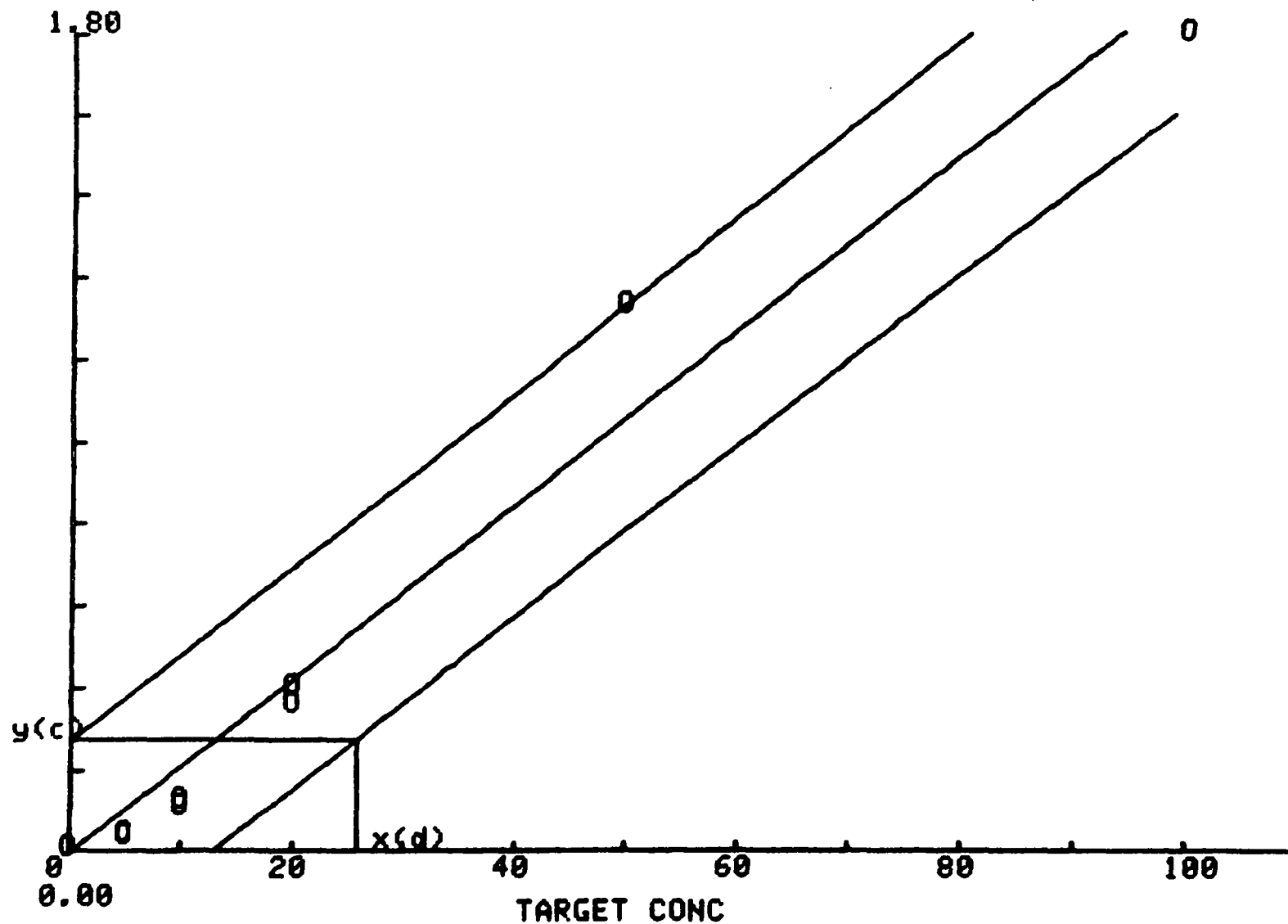
t= 1.81245868646

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.245604514821

x(d)= 25.8249834303

NITROGLYCERIN IN WATER  
RUNS 1 2  
ST PEAK AREA



VERTICAL AXIS TIC INTERVAL = 0.18

PETH IN WATER

RUNS 1 2

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. MG/L	Day 1 Found Conc. MG/L	Day 2 Found Conc. MG/L	Day 3 Found Conc. MG/L	Day 4 Found Conc. MG/L
0.00	0.13	0.00		
5.00	5.33	5.00		
10.00	9.24	10.00		
20.00	20.30	20.00		

PETH IN WATER

RUNS 1 2

TARGET CONC

ANALYSIS OF 8 TARGET CONC-PEAK AREA POINTS

MEAN= 8.75 SD= 7.90569415042

PEAK AREA

MEAN= 0.1375 SD= 0.1164658135

N0. RUNS 2 TOTAL X-Y ALL RUNS 8 N0. CONCENTR 8

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.009

SLOPE= 0.0146857142857

R= 0.996865621198

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 9.904761905E-5

COMPUTE T

D.F.= 6

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

t= 1.94317596321

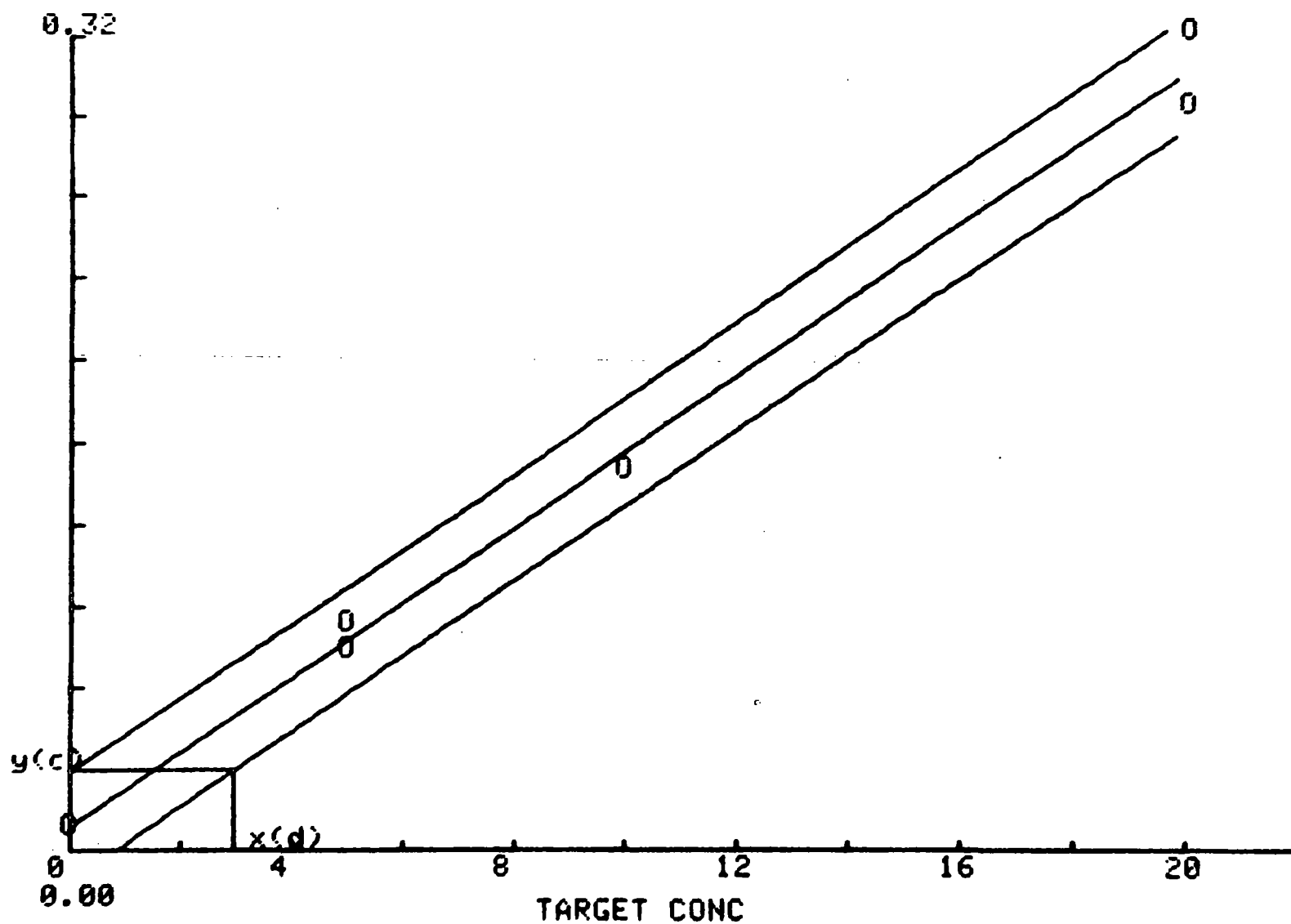
1

REPLICATES ON UNKNOWN SAMPLE 1

y(c)= 0.0310498594706

x(d)= 2.94522299778

PETH IN WATER  
RUNS 1 2  
PEAK AREA



VERTICAL AXIS TIC INTERVAL = 0.032

PETH IN WATER

RUNS 1 2

COMPILATION OF TARGET CONC. VS PEAK AREA

Target Conc. MG/L	Day 1 Found Conc. MG/L	Day 2 Found Conc. MG/L	Day 3 Found Conc. MG/L	Day 4 Found Conc. MG/L
0.00	-13.45	-12.45		
5.00	0.26	-1.01		
10.00	10.55	10.42		
20.00	39.69	33.28		
50.00	53.40	64.31		
100.00	94.54	90.45		

PETH IN WATER  
RUNS 1 2  
TARGET CONC  
ANALYSIS OF 12 TARGET CONC-PEAK AREA POINTS

MEAN= 30.8333333333 SD= 36.4837032942

PEAK AREA  
MEAN= 0.271666666667 SD= 0.227669350887

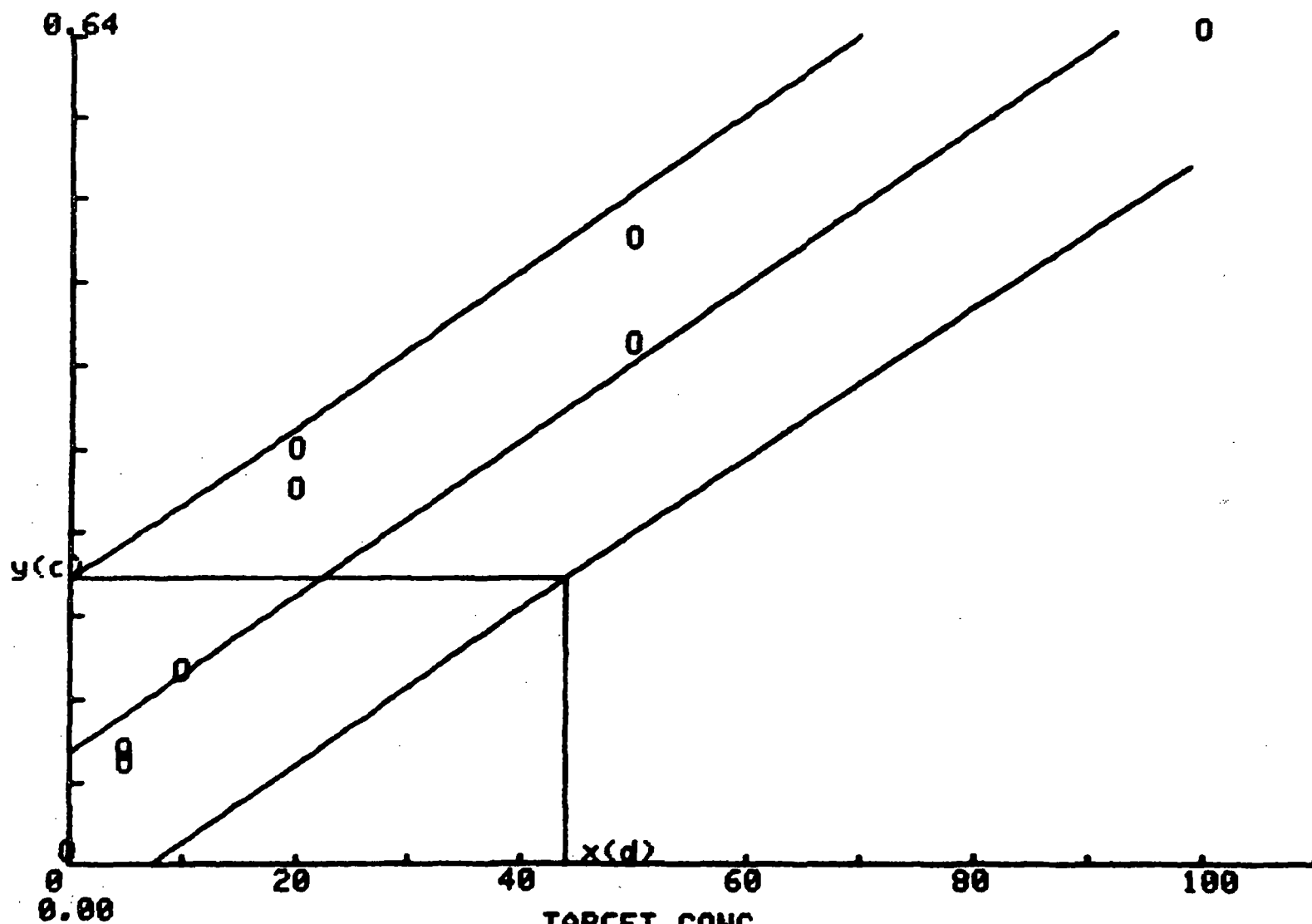
N0. RUNS 2 TOTAL X-Y ALL RUNS 12 N0. CONCENTR 12  
MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= 0.0873335230507  
SLOPE= 0.00597837222538  
R= 0.958026003955  
MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00468598178714  
COMPUTE T

D.F.= 10  
ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS  
.05 SO TOTAL P= .1)

.1  
t= 1.81245868646  
REPLICATES ON UNKNOWN SAMPLE 1  
y(c)= 0.220283817974  
x(d)= 43.9748342347

PETN IN WATER  
RUNS 1 2  
PEAK AREA

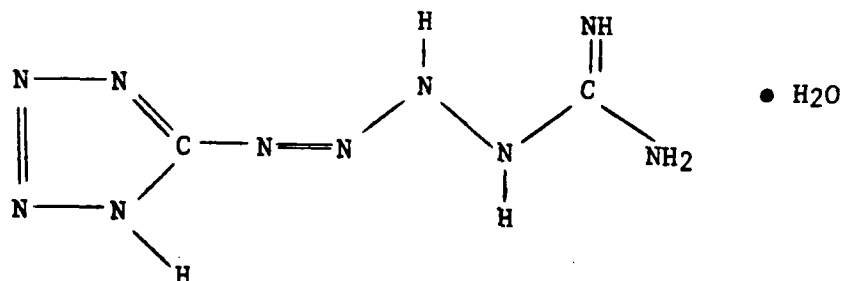


VERTICAL AXIS TIC INTERVAL= 0.064

DETERMINATION OF TETRAZENE IN WATERI. APPLICATION

This method is a quantitative procedure for determining tetrazene in water.

- A. Tested Concentration Range. The tested concentration range is from 500 to 10,000  $\mu\text{g/l}$ .
- B. Sensitivity Limits. At the detection limit,  $1 \text{ ng} = 2.5 \times 10^{-4} \text{ AU}$ .
- C. Detection Limits. (Calculated using the method of Hubeaux and Vox). Detection limit is 500  $\mu\text{g/l}$  (lowest level standard).
- D. Interferences. Compounds containing a guanidine moiety will interfere.
- E. Analysis Rate. Approximately twenty samples can be analyzed by a worker in an eight hour day.

II. CHEMISTRYStructure

CAS RN # --

Melting point -- decomposes

Boiling point -- N/A

Handling hazards -- Tetrazene is explosive especially in the dry state. The neat solid should be stored in isopropanol and only a small portion should be dried, by placing on a clean filter paper, to prepare analytical standards.

### III. APPARATUS

- A. Instrumentation. Bausch and Lomb Spectronic 20 spectrometer with tungsten lamp.
- B. Parameters. Set spectrometer at 530 nm. Instrument zero is set using a blank sample with each set of samples.
- C. Hardware/Glassware.
  - 1. Volumetric flasks (various sizes).
  - 2. Pipets 10 ml and 1 ml.
  - 3. 4 dram vials with polyseal caps.
  - 4. 100 ml graduated cylinder.
  - 5. 8 oz screw capped bottles with Teflon lined caps.
  - 6. Cuvettes for Spectronic 20.
  - 7. Millipore all glass filter apparatus.
  - 8. Millipore filters, 0.45 $\mu$ , type HA, 47mm diameter.
- D. Chemicals.
  - 1. Methanol, distilled in glass, Burdick and Jackson.
  - 2. Acetone, distilled in glass, Burdick and Jackson.
  - 3. Distilled water, Millipore "Millie Q" or equivalent.
  - 4. Sodium nitroprusside, analytical reagent grade.
  - 5. Potassium ferricyanide, analytical reagent grade.
  - 6. Sodium hydroxide, analytical reagent grade.
- E. Reagents
  - 1. Sodium Hydroxide. 10 grams in 100 ml distilled water. Store refrigerated in an eight ounce screw capped bottle.
  - 2. Sodium Nitroprusside. 10 grams in 100 ml distilled water. Store refrigerated in a 8 ounce screw capped bottle. Prepare fresh every two weeks.
  - 3. Potassium Ferricyanide. 10 grams in 100 ml distilled water. Store refrigerated in a 8 ounce screw capped bottle. Prepare fresh every two weeks.
  - 4. Color Developing Reagent. Mix 10 ml portions of reagents A, B, and C with 30 ml of distilled water in a 125 ml flask. Allow to stand for 20 minutes and then mix with 60 ml of acetone. Use reagent within one hour.

#### IV. STANDARDS

##### A. Calibration Standards.

Stock. Place 10 mg of tetrazene in 100 ml volumetric flask. Fill to mark with methanol and mix well. Store refrigerated in a Teflon lined 8 oz bottle.

Working. Prepare calibration standards containing 0, 1, 2.5, 5, and 10 mg/liter in water by placing 0, 1, 2.5, 5 and 10 ml of the stock in a series of 100 ml volumetric flasks and filling to the mark with water.

##### B. Control Spikes. Since no extraction is involved control spikes are equivalent to working standards.

#### V. PROCEDURE

A. Sample Preparation. Obtain at least 50 ml of each water sample and filter through a millipore filter. Place exactly 10 ml of the filtered water sample in a screw capped vial and store refrigerated until analyzed.

B. Color Development. Pipet 1 ml of freshly prepared color developing reagent into 10 ml of the water sample, mix, and let stand for 1 hour. Run process blank and calibration standards in the same manner.

C. Sample Analysis. Set Spectronic 20 at 530 nm and allow the instrument to warm up for at least 30 minutes. Set 0 absorbance using the process blank sample. Measure the absorbance of each sample and calibration standard.

#### VI. CALCULATION

Construct a calibration curve from the data (concentration vs. absorbance) obtained for calibration standards and interpolate the concentration in the sample from the calibration curve. (Above 5 ppm the curve will probably be nonlinear).

Spike Recovery Data From Water

<u>Target Value</u>	<u>Found Value</u>
0	0, -0.005, +0.05, +0.05
0.5	0.5, 0.5, 0.48, 0.53
1.0	1.0, 1.0, 0.98, 1.0
2.5	2.5, 2.4, 2.6, 2.5
5.0	5.0, 4.8, 4.9, 4.9
10.0	10.0, 10.0, 9.9, 10.1

VII. REFERENCE. None

**BCL TETRACENE IN WATER P & A****RUNS 1 2 3 4****COMPILATION OF TARGET CONC. VS FOUND CONC**

<b>Target Conc. MG/L</b>	<b>Day 1 Found Conc. MG/L</b>	<b>Day 2 Found Conc. MG/L</b>	<b>Day 3 Found Conc. MG/L</b>	<b>Day 4 Found Conc. MG/L</b>
<b>0.00</b>	<b>0.00</b>	<b>0.00</b>	<b>0.05</b>	<b>0.05</b>
<b>0.50</b>	<b>0.50</b>	<b>0.50</b>	<b>0.48</b>	<b>0.53</b>
<b>1.00</b>	<b>1.00</b>	<b>1.00</b>	<b>0.98</b>	<b>1.00</b>
<b>2.50</b>	<b>2.50</b>	<b>2.40</b>	<b>2.60</b>	<b>2.50</b>
<b>5.00</b>	<b>5.00</b>	<b>4.80</b>	<b>4.90</b>	<b>4.90</b>
<b>10.00</b>	<b>10.00</b>	<b>10.00</b>	<b>9.90</b>	<b>10.10</b>

BCL TETRACENE IN WATER P & A

RUNS 1 2 3 4

TARGET CONC

ANALYSIS OF 24 TARGET CONC-FOUND CONC POINTS

MEAN= 3.16666666667 SD= 3.54678870881

FOUND CONC

MEAN= 3.15375 SD= 3.53473055673

NO. RUNS 4 TOTAL X-Y ALL RUNS 24 NO. CONCENTR 24

MEASURES (Y'S) EACH TARGET CONC 1

INTERCEPT= -0.0016071428571

SLOPE= 0.996428571429

R= 0.999827723656

MEAN SQR DEV OF POINTS FROM REGRESSION (ST ERROR EST)= 0.00450024350701

COMPUTE T

D.F.= 22

ENTER 2 TAIL P LEVEL (USUALLY .1, EACH CONFID BAND IS

.05 SO TOTAL P= .1)

.1

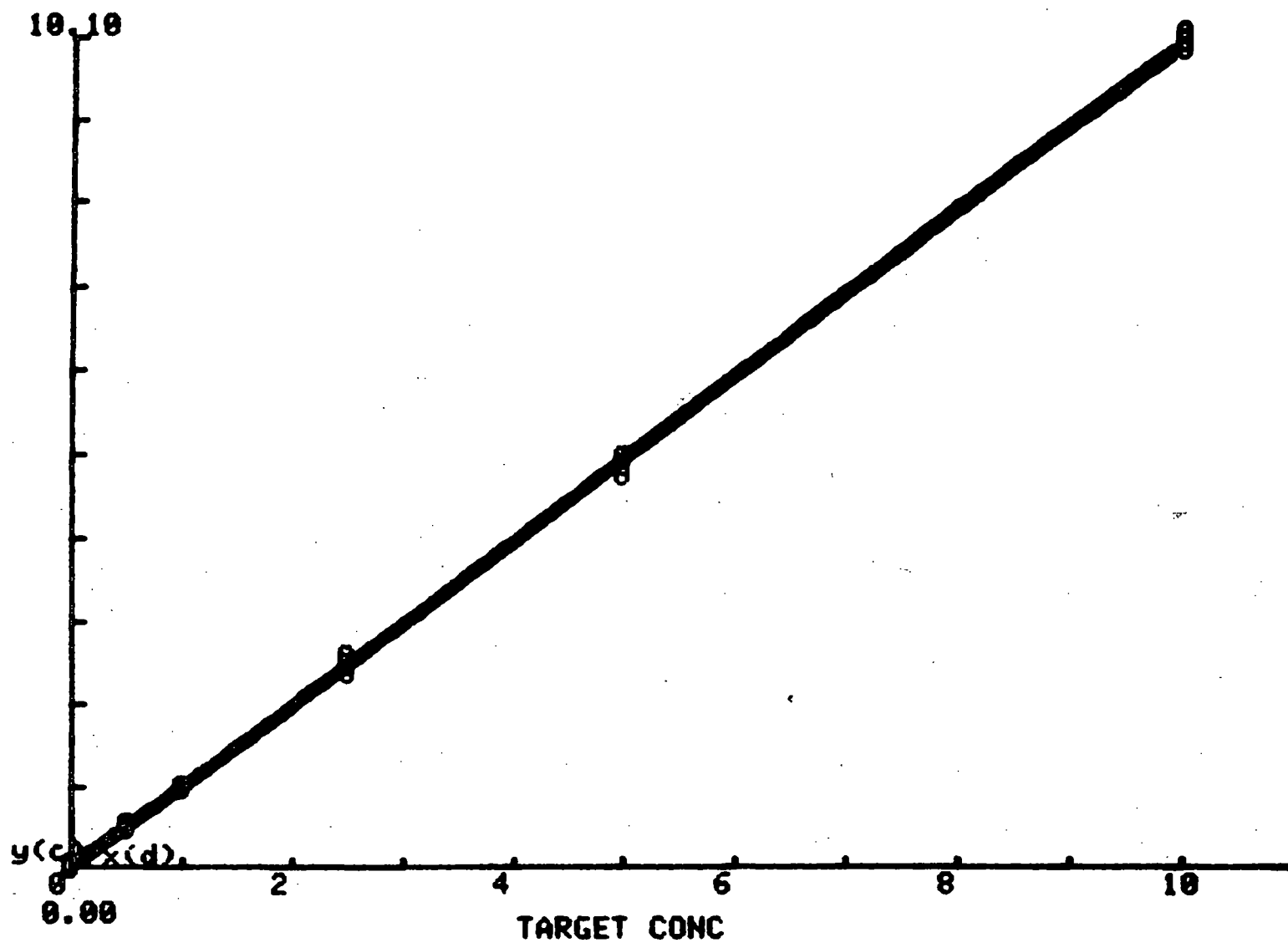
t= 1.71713909197

REPLICATES ON UNKNOWN SAMPLE 4

y(c)= 0.0641962943042

x(d)= 0.121792130263

BCL TEIRACENE IN WATER P & A  
RUNS 1 2 3 4  
FOUND CONC



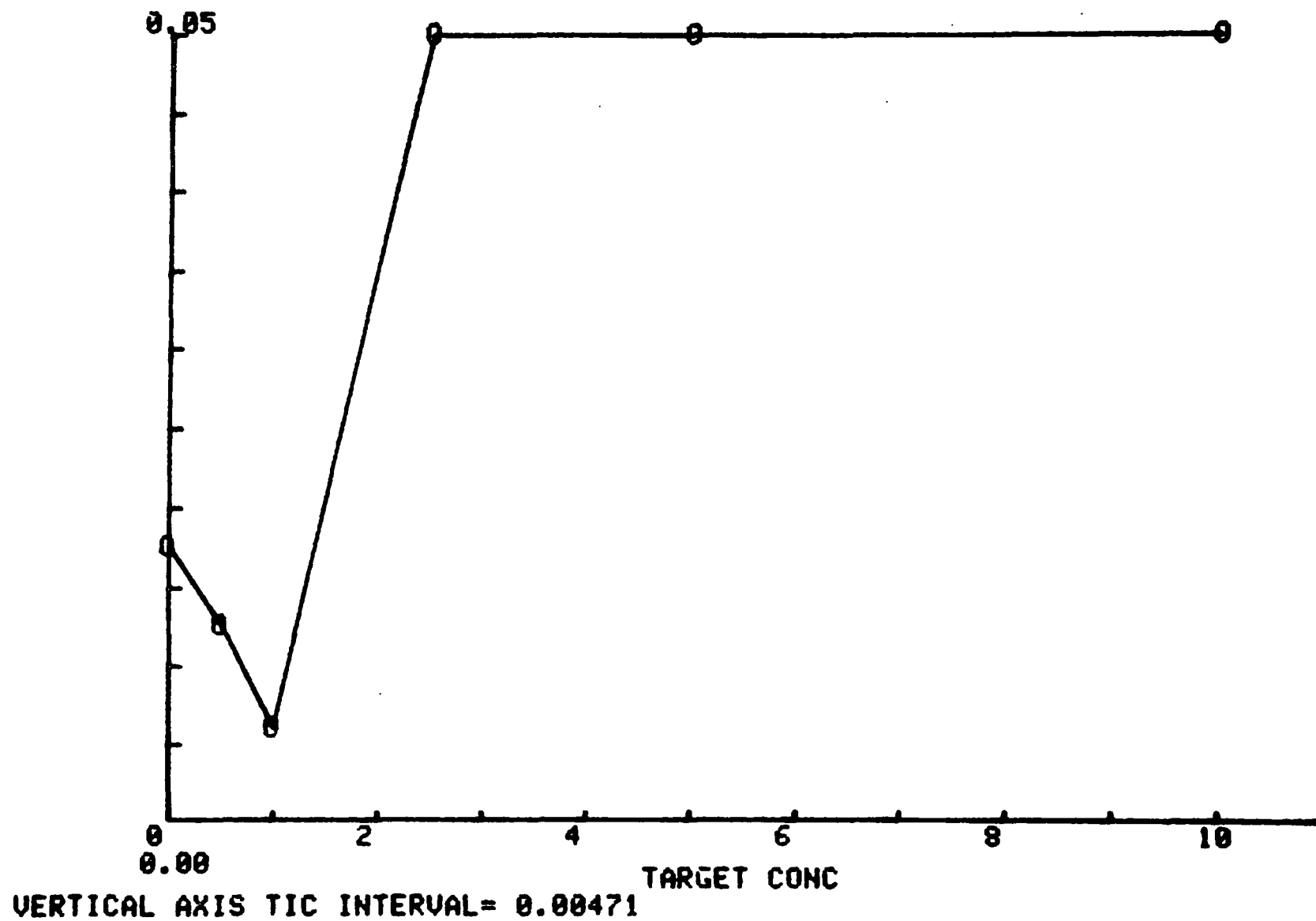
**BCL TETRACENE IN WATER P & A**

RUNS 1 2 3 4

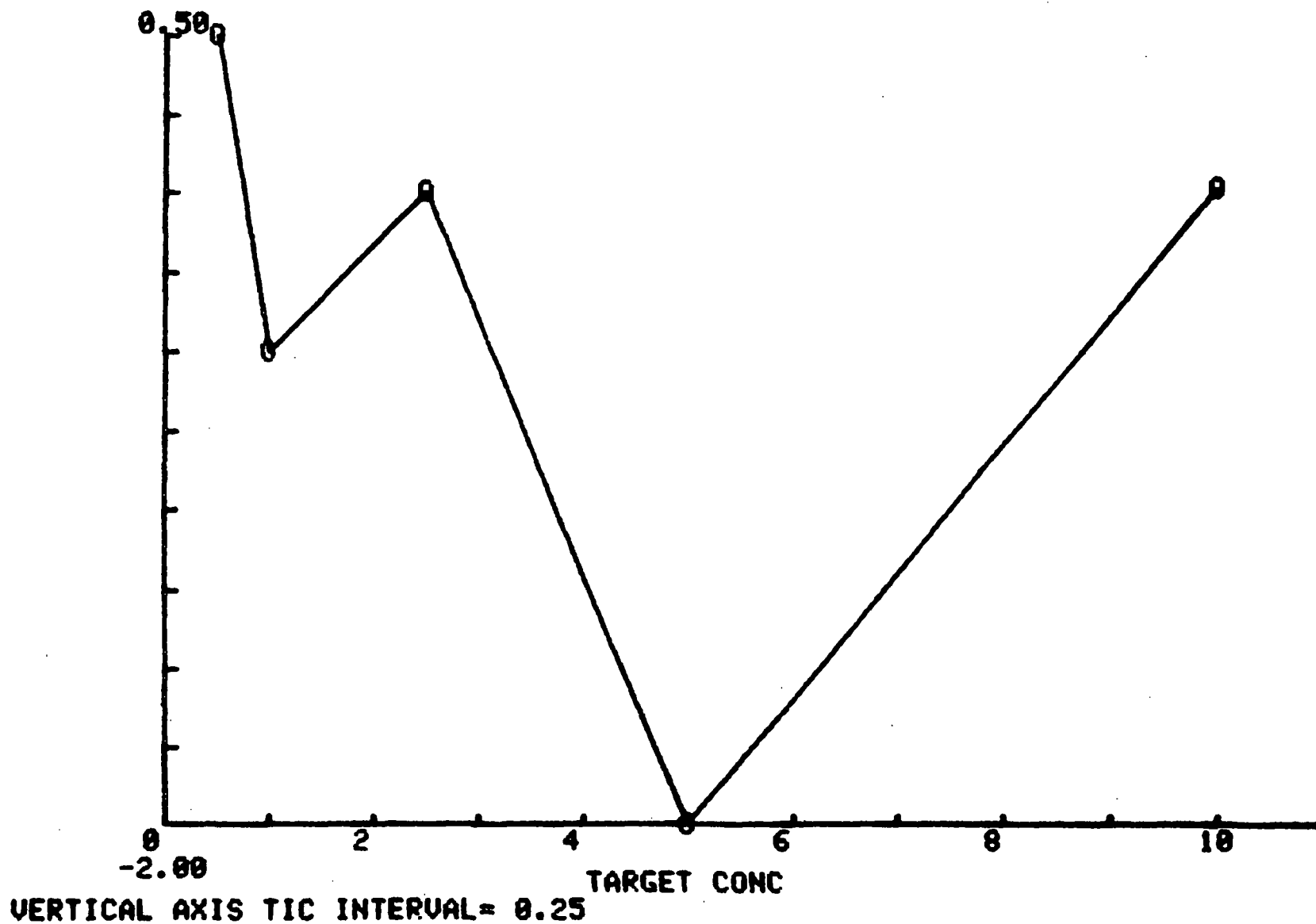
**STATISTICAL DATA USED TO DETERMINE PERCENT  
INACCURACY AND COEFFICIENT OF VARIATION**

Target Conc. MG/L	Mean Found Conc. MG/L	Standard Deviation	Percent Inaccuracy	Coefficient(%) of variation
0.00	0.03	0.02		66.67
0.50	0.50	0.01	0.50	2.37
1.00	1.00	0.01	-0.50	0.58
2.50	2.50	0.05	0.00	1.89
5.00	4.90	0.05	-2.00	0.96
10.00	10.00	0.05	0.00	0.47

BUL TETRACENE IN WATER P & A  
RUNS 1 2 3 4  
STANDARD DEVIATION



BCL 1,2,3,4 IN WATER P & A  
RUNS 1 2 3 4  
MEAN PERCENT INACCURACY



STANDARD DEVIATION  
BCL TETRACENE IN WATER P & A  
RUNS 1 2 3 4  
USE FOUND CONC  
(1) 0 0.0166 (2) 0.5 0.0119  
(3) 1 0.0057 (4) 2.5 0.0471  
(5) 5 0.0471 (6) 10 0.0471

INDIVIDUAL PERCENT INACCURACY  
BCL TETRACENE IN WATER P & A  
RUNS 1 2 3 4  
USE FOUND CONC

(1) 0 1.0E+300 (2) 0.5 0

(3) 1 0 (4) 2.5 0

(5) 5 0 (6) 10 0

(7) 0 1.0E+300 (8) 0.5 0

(9) 1 0 (10) 2.5 -4.0001

(11) 5 -4.0001 (12) 10 0

(13) 0 1.0E+300 (14) 0.5 -4.0001

(15) 1 -2.0001 (16) 2.5 3.9999

(17) 5 -2.0001 (18) 10 -1.0001

(19) 0 1.0E+300 (20) 0.5 5.9999

(21) 1 0 (22) 2.5 0

(23) 5 -2.0001 (24) 10 0.9999

COMPUTE MEAN PERCENT INACCURACY? Y/N Y

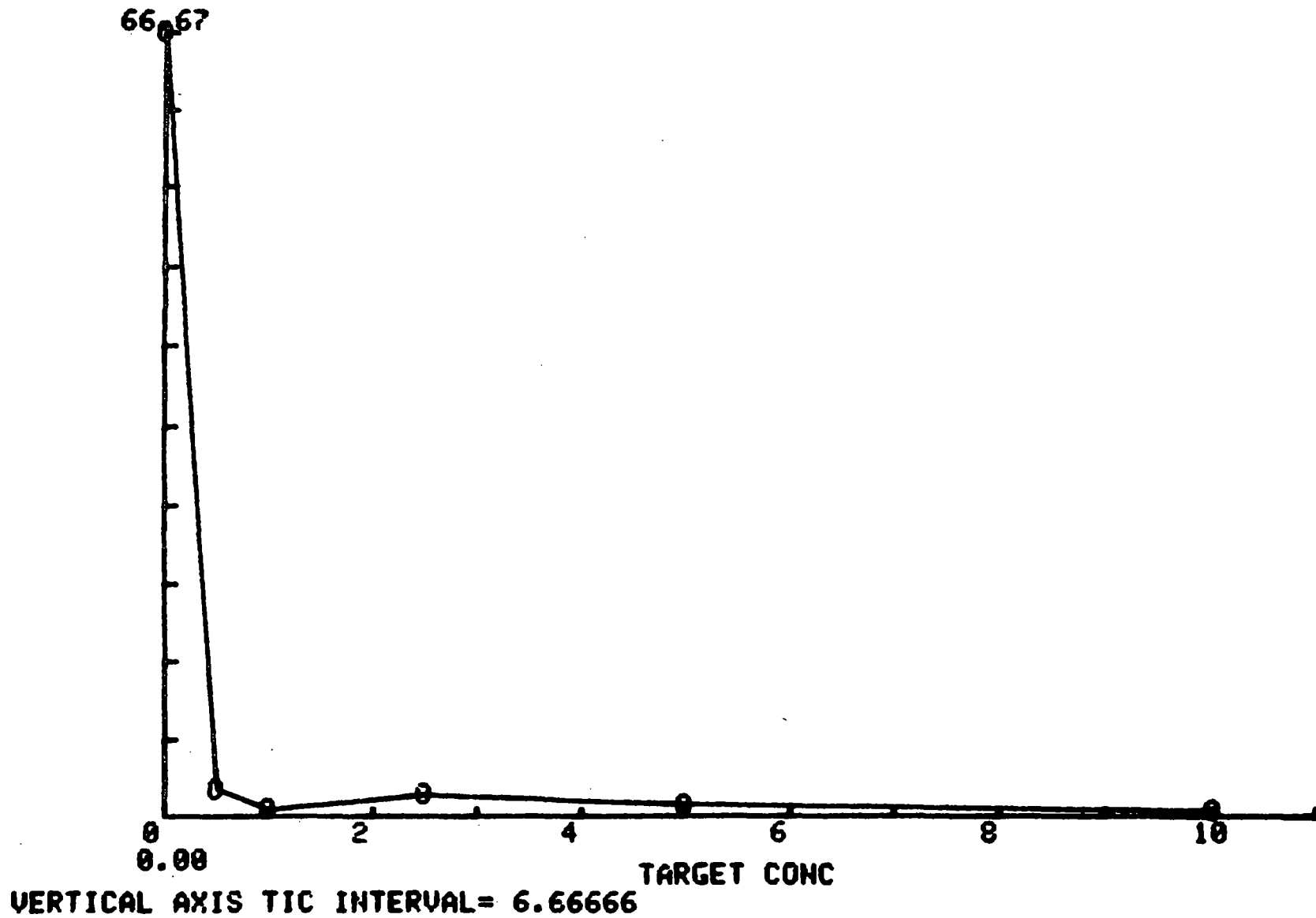
(1) 0 1.0E+300 (2) 0.5 0.4999

(3) 1 -0.5001 (4) 2.5 -1.0E-4

(5) 5 -2.0001 (6) 10 -1.0E-4

PERCENT IMPRECISION  
BCL TETRACENE IN WATER P & A  
RUNS 1 2 3 4  
USE FOUND CONC  
(1) 0 66.6666 (2) 0.5 2.3686  
(3) 1 0.5802 (4) 2.5 1.8856  
(5) 5 0.962 (6) 10 0.4714

BCL 1,2-DICHLOROBENZENE IN WATER P & A  
RUNS 1 2 3 4  
PERCENT IMPRECISION



**APPENDIX G**

**SAMPLE STANDARD OPERATING PROCEDURE**

**"Demolition Plan for the 400 Area  
(Explosives Manufacturing Area) of the  
Frankford Arsenal**



Rockwell International  
Atomic International Division

## SUPPORTING DOCUMENT

NUMBER

N5050P000001

REV LTH/CHG. 1.3

PROGRAM TITLE

Frankford Arsenal Decontamination/Cleanup  
Operations

DOCUMENT TITLE

Demolition Plan for the 400 Area (Explosives  
Manufacturing Area) of the Frankford Arsenal

DOCUMENT TYPE

Operating Procedure

KEY NOUNS

Demolition, Decontamination, Frankford Arsenal

ORIGINAL ISSUE DATE

GO NO.

08505

S/A NO

21000

PAGE 1 OF

TOTAL PAGES 22

REL. DATE

PREPARED BY/DATE

W. R. Johnson

DEPT

731

MAIL ADDR

T055

F. C. Schrag

731

T020

IR&D PROGRAM? YES ☐

NO ☒

IF YES, ENTER TPA NO.

APPROVALS

A. F. Lillie

R. Brengle

W. R. McCurnin

DATE

12/19/79

12/19/79

12/19/79

SECURITY CLASSIFICATION

(CHECK ONE BOX ONLY)

UNCL

DOE

DOD

CONF.

SECRET

☒

☐

☐

☐

(CHECK ONE BOX ONLY)

RESTRICTED

DATA

DEFENSE

INFO

☐

☐

AUTHORIZED  
CLASSIFIER

DATE

DISTRIBUTION

* NAME	MAIL ADDR
* A. J. Darnell	NB04
* D. McClelland	LB13
* J. Miller	T020
* R. Tuttle	NB13
* A. F. Lillie (2)	LB05
* R. D. Gothar (7)	LA38
* A. J. Lucas	LA34
* J. C. Campbell	KB07
* W. W. Kelly	KA02
* M. Murray	KA46
* R. G. Brengle	LB08
* W. R. McCurnin	T020
* J. H. Walter	T006
* D. McKenzie	NB04
* G. H. Hallinan	LB08
* W. D. Kittinger	NB02
* G. W. Meyers	LA10
* G. Colovos (2)	EMSC

ABSTRACT

Presented is a step-by-step procedure for the demolition of the 400 area of the Frankford Arsenal. There are 32 buildings, covered walkways, sumps, sewers, and concrete blast walls which will be removed. The concrete floors and macadam paving will also be removed. Below-grade foundations, pilings, and the concrete roadway are not to be removed. The excavated area will be graded. Only three of the buildings are slightly contaminated with explosive residue as reported by Battelle in Appendix A of Contract DAAK 11-79-C-0135. Included in this procedure is a list of the buildings along with the type of contaminate reported. The area is to be restored to a condition which will allow its unrestricted use (so-called 5X decon).

RESERVED FOR PROPRIETARY/LEGAL NOTICES

716-M.90/sjd

\* COMPLETE DOCUMENT

NO ASTERISK, TITLE PAGE SUMMARY  
OF CHANGE PAGE ONLY

## CONTENTS

	<u>Page</u>
1.0 SCOPE.....	3
2.0 BACKGROUND.....	3
3.0 METHODS.....	4
4.0 CONTAMINANTS AND MATERIALS INVOLVED.....	6
5.0 EQUIPMENT AND MATERIALS.....	7
6.0 SAFETY PROCEDURES.....	8
7.0 PROCEDURES FOR DEMOLITION.....	9
8.0 COST AND SCHEDULE.....	11

## FIGURES

<u>Figure</u>	<u>Page</u>
1. Map of Frankford Arsenal.....	12
2. Concept Demolition of 400 Area.....	17
3. Demolition Sequence.....	19
4. Remote Flame Operation.....	22
5. Typical Blast Wall Cross Section.....	23

## TABLES

<u>Table</u>	<u>Page</u>
1. Manufacturing Operations in the 400 Area.....	13
2. 400 Area Buildings.....	15
3. Thermal Decomposition Temperatures of Selected Explosives...	20
4. 400 Area Sump and Vent Contamination.....	21

APPENDIX A: RESTRICTED ACCESS AREA ENTRY PERMIT.....	24
------------------------------------------------------	----



## 1.0 SCOPE

This procedure will provide for the safe demolition of structures in the 400 area of the Frankford Arsenal. All structures will be removed to the existing grade level and all materials will be removed. Surfaces potentially contaminated with explosives will be flamed to assure their cleanliness. The area will be restored for unrestricted use.

## 2.0 BACKGROUND

The 400 area is a 9-acre section of Frankford Arsenal (FFA) located in the southeast end of the post. The 400 area was used to manufacture primer mixes and pyrotechnic materials. Lead styphnate manufacture was started in 1944, and production continued to 1976. Table 1 is a tabulation of the manufacturing operations for each of the buildings.

The area includes 32 one- and two-room buildings, blast walls, associated drains, traps, sumps, and approximately 8,200 ft of terra cotta drain lines. The drain lines include branch waste lines from each building's sump which connect with the main 30-in. line running to the Delaware River. In addition, the old Fidler sewer line to the Delaware River runs adjacent to the 30-in. sewer line. Wash-down of process areas and decanting of vessels during operations contributed extensively to contamination of the sumps and laterals. The Battelle survey (Appendix A of Contract DAAK 11-79-C-D135) revealed that Buildings 404, 410, and 429 contained explosive residues. Some of the sumps, drains, and vents associated with the buildings were also found to contain some explosive residues.

The 400 area is shown in Figure 1 (Drawing FF 12958 enlargement, "Map of Frankford Arsenal"). A tabulation of the buildings and associated structures is presented in Table 2. Included in the table is the type of explosive contamination reported in the Battelle survey (Appendix A of Contract). Detailed drawings of the 400 area are on file at the Arsenal and are in the FF 14000 series.



### 3.0 METHODS

A conceptual process flow diagram for the demolition of the 400 area is presented in Figure 2. Process details are presented in Section 7.

The demolition for the 400 area consists of a scoping survey of the area for explosives to verify the Battelle results. The wooden covered walkways, along with the asbestos shingles, will be removed and treated as asbestos waste. The wooden roof of each building will be removed along with the asbestos shingles and asbestos ceiling materials. This mass of material will be treated as asbestos contaminated waste and processed for burial. The land burial facility will be GROWS, Geological Reclamation Operation and Waste Systems, Inc., Morrisville, Pennsylvania. The inside of the buildings, which include the concrete floor, floor tiles, and ceramic tile walls will be flamed. The concrete blast walls will be removed and processed to a land fill area. The buildings will be demolished and the materials will be removed and processed to a land fill area. The drains and sumps will be removed; also the concrete floor from each of the buildings will be removed and processed for land fill. The macadam paving between the buildings will be excavated and removed for land fill. The area will be backfilled with soil and final-surveyed for explosive contaminants before releasing the area for unrestricted use. After the area has been released, the perimeter fence will be removed and disposed of as scrap.

Flaming will be used for noncombustibles on the inside of the buildings and sumps to assure that all residue explosives are decomposed. Flaming will be done to raise the surface temperature of structures above the decomposition temperature of the explosive contaminants. Temperature indicating paints will be used to assure that adequate reaction temperatures are obtained. Table 3 lists the explosives which were manufactured in the 400 area, and their decomposition temperatures. Before demolition of the buildings, the vents will be removed.



After flaming, typical surfaces of Building 429A will be sampled with swipe wet with acetone. These samples will be analyzed for explosive residue as a double-check to assure that the flaming is effectively decomposing explosives.

Some of the sumps may have radioactive contamination as reported by Battelle. Table 4 gives a listing of the sumps that may be contaminated. Samples will be taken from these sumps to verify the presence of radioactive contamination. Those sumps that do not contain radioactive contamination will be treated as only containing explosive contamination. Those sumps that do have radioactive contamination will be treated for explosive contamination and then treated for radiological materials.

A few of the sumps may have heavy metal residue as reported by Battelle. These sumps will be sampled for heavy metal contamination. Those sumps that do, in fact, have heavy metal present will be treated for explosive contamination and then cleaned of heavy metal if it is still above allowable limits before they are flamed as part of the explosive residual cleanup operation.

The sumps will be pumped to remove liquid and sludge. This waste will be thermally or chemically treated to decompose the explosive residues. Flaming of the sumps will be performed before removal to assure that all explosive residue have been reacted. Temperature indicating paints will be used to assure that adequate reaction temperatures are obtained. The drains will be removed, then passed through a flame to react explosive residue. The materials from the drains and sumps will be transported to a land fill site.

Waste collected in vacuum cleaners and miscellaneous potentially contaminated materials such as work gloves, wipes, etc., will be flashed to decompose explosive residues.



A single designated working copy of this demolition plan may be changed in red (red lines) by authorized personnel as procedural changes dictate. All procedural changes must be approved by a U.S. Army Toxic and Hazardous Material Agency (USATMA) representative. These include, but are not limited to:

- a) Health and Safety Engineering Coordinator (HSEC) must approve and sign those changes affecting health and safety.
- b) The site director must approve and sign changes affecting the scope of the task, i.e., cost or schedule.
- c) QA must approve and sign those changes affecting quality.
- d) All changes must be approved by D&D operations.

Waste materials will be controlled by D&D operations management. Only haulers with state permits will be used to transport waste. Traffic will initiate material removal requests which must be approved by operations management. Each vehicle entering and leaving will be checked to assure that only materials approved on the removal request are in the vehicle and that the delivery destination is correct.

#### 4.0 CONTAMINANTS AND MATERIALS INVOLVED

There are some contaminants and materials involved in the demolition of the 400 area that require special attention. The Battelle survey (Appendix A of the contract) reports explosive residue contamination in a few of the buildings in the 400 area. Table 2 lists the contamination reported by Battelle in the 400 area buildings. Table 3 lists the decomposition temperature for these explosives. Table 4 lists the contamination reported by Battelle in the 400 area support facilities.

For the inside of buildings having a history of explosive usage (see Table 1), a battery of five spray indicator tests will be used to screen for explosives. These spray tests are extremely useful since a large



number of surfaces can be screened rapidly. However, it should be noted that a positive result must be confirmed using a more specific assay method, since all tests of this type are subject to false positives.

Along with the explosive residues, the asbestos materials will require special handling. Asbestos materials are limited to the roofs of buildings and walkways, pipe insulation, and ceilings of all buildings. The roof tiles and transite ceiling materials are hard and fired materials which will not produce an airborne hazard. The pipe insulation is friable and may produce loose fibers. Workers will be protected from this hazard by respirators and protective clothing.

#### 5.0 EQUIPMENT AND MATERIALS

The equipment required for demolition shall include, but not be limited to, the following:

- 1) Caterpillar - to remove walkways, demolish buildings
- 2) Wrecking ball - to demolish blast walls
- 3) Hiram - to demolish divider walls, floors
- 4) Front loader - to remove macadam, remove rubbish
- 5) Backhoe - to remove sewers, sumps
- 6) Remote boom/with torch - to flame inside buildings
- 7) Vacuum cleaner (nonsparking, air-powered)
- 8) Respirators
- 9) Coveralls (nonstatic cotton)
- 10) Safety shoes (with grounding shoe straps)
- 11) Hard hats
- 12) Safety glasses.



## 6.0 SAFETY PROCEDURES

Limited access to the 400 area will be maintained during all operations of demolition. Personnel shall be limited to those essential for the task; however, there shall be no less than two persons while working in or around contaminated areas.

A Restricted Access Area Entry Permit (Appendix A) initiated by Operations and issued by the Health and Safety Radiation Services will be required for all work performed in the 400 area. This permit will include procedures outlining minimum safety requirements for these hazardous areas and operations. Following review, the permit shall be signed by all personnel who are doing work covered by the permit. A copy of the permit will be posted in the work area. No entry shall be permitted without specific approval of the supervisor in charge of the area.

Air-powered, nonsparking vacuum equipment will be used for cleanup operations. The vacuum equipment will be connected to building electrical ground when operated in areas containing explosive residues. The vacuum cleaner attachments will be made of nonsparking materials, such as aluminum, and will be grounded to the vacuum cleaner. The vacuum equipment will be operated with sufficient water in the collection tank to keep the residue wet or moist at all times. The vacuum equipment will be equipped with special exhaust filters to reduce discharge of particulates in the atmosphere. Nonsparking tools will be used in areas contaminated with explosive residue. Grounding shoe straps and nonstatic cotton coveralls will be worn by personnel in cleaning up explosives residue.

The flaming operation and cleanup operations will be performed with caution when in the proximity of combustible materials. Appropriate fire prevention measures will be exercised. Fire retardant clothing will be worn by the operators during flaming operations.



All equipment operators will be required to wear eye protection and hard hats. All nonequipment operators will be required to wear the same, with the addition of safety shoes. Steel plates will be added to equipment as armor to protect the operators.

Special note should be taken for the existence of explosives residue in cracks and crevices. If such residues are found, appropriate removal procedures such as steam cleaning or high-pressure water washing techniques will be employed.

#### 7.0 PROCEDURES FOR DEMOLITION

The following procedures are outlined for performing the demolition of the 400 area: (re: Figure 3).

- 1) Remove steel beams and steam pipe holders along walkway. Beams and pipes are to be processed as scrap metal.
- 2) Remove pipes from covered walkway. Process pipes as scrap metal. If asbestos insulation is present, the insulation and the enclosed pipe will be disposed as asbestos waste.
- 3) Remove covered walkways and collect all materials in one area before moving it to a landfill.
- 4) Removal of building roofs.
  - a) Survey all interior surfaces of the roof with spray indicator.
  - b) Vacuum and hand scrub surfaces that indicate presence of explosive contamination.
  - c) Remove roof from each building - mechanized removal. Collect all materials in one area.
  - d) Remove all materials from site and handle as asbestos waste for land burial.
- 5) Removal of buildings.



- a) Remove all loose combustible materials from all buildings.
  - b) Survey the walls and floors with spray indicators. Only those buildings or portions of those buildings which contain explosive residue as shown by the indicator will be cleaned by vacuum techniques. Resurvey the area to assure that the area of the building that previously indicated the presence of explosive residue is clean to less than  $1 \text{ gm/m}^2$  ( $0.1 \text{ gm/m}^2$  for nitroglycerin).
  - c) For areas of the buildings which continue to indicate the presence of explosive residue, additional cleaning will be done. Methods such as steam cleaning, washing/scrubbing or foam cleaning either separately or in combination will be used.
  - d) Verification to show the explosives residue has been reduced to a safe level in all buildings. If not, Procedure 5c will be repeated until analyses shows that a safe level has been reached.
- 6) Flame inside of buildings by remote boom torch. Temperature indicating paints will be used to show that adequate reaction temperatures are obtained. The operation will be monitored by TV and the torch operator will be protected by steel armor (Figure 4).
- 7) Verification sampling of typical flamed surfaces will be done to confirm that the explosives residue have been decomposed.

Materials from the following procedure steps will be collected, then processed to a land fill.

- 8) Demolish blast walls (typical blast wall shown in Figure 5).



- 9) Demolish buildings with bulldozer.
- 10) Demolish divider walls and concrete buildings.
- 11) Remove floors of buildings.
- 12) Remove sumps after:
  - a) Pump the liquids out.
  - b) Flame the inside of the sump using temperature indicating paints to show that a high enough temperature was reached to decompose explosives residue.
- 13) Remove sewers.
  - a) Pass all sewer material through flame.
  - b) Use temperature indicating paint on some selective surfaces to assure that high enough temperatures were reached to decompose explosives residue.
- 14) Backfill sump and sewer line excavation with soil.
- 15) Remove macadam.
- 16) Rough grading of 400 area to make level.

#### 8.0 COST AND SCHEDULE

Demolition of the 400 area is planned for early summer after the ground has defrosted. Excavation will be much easier and liquids easier to manage after the winter freeze is over.

Cost estimates for demolition of the 400 area will be obtained from demolition contractors at part of Phase III of this contract. Current plans are to subcontract the work of the demolition phase.



FIGURE 2  
CONCEPT DEMOLITION OF 400 AREA  
(Continued)





FIGURE 3  
DEMOLITION SEQUENCE

<u>EQUIPMENT</u>	<u>TASK</u>	<u>DEPOSITION</u>
HAND AND TORCH	REMOVE STEEL BEAMS AND STEAM HOLDERS	SCRAP
HAND	REMOVE PIPES FROM COVERED WALK	SCRAP
CAT	REMOVE COVERED WALKS	BURY AS ASBESTOS LAND BURIAL
REMOTE WITH CLAM SHELL	REMOVE ROOF FROM EACH BUILDING	RUBBLE PILE ON SITE      AGE      BURY AS ASBESTOS LAND BURIAL
HAND	REMOVE DOOR AND WINDOW FRAMES	RUBBLE PILE ON SITE      BURY LAND BURIAL
REMOTE BOOM	FLAME INSIDE OF BUILDING TORCH	
WRECKING BALL HYRAM	DEMOLISH BLAST WALLS	LANDFILL
BULLDOZER	DEMOLISH BUILDINGS	LANDFILL
HYRAM	DEMOLISH DIVIDER WALL AND CONCRETE BUILDINGS	LANDFILL
HYRAM	REMOVE FLOORS	LANDFILL
	PUMP LIQUIDS FROM SUMPS	
	FLAME THE INSIDE OF SUMPS	
FRONT LOADER	REMOVE SEWERS	LANDFILL
	FLAME SEWER MATERIALS	
BACK HOE	REMOVE SUMPS	LANDFILL
HYRAM	BACKFILL WITH SOIL	



TABLE 3  
THERMAL DECOMPOSITION TEMPERATURES OF SELECTED EXPLOSIVES\*

Explosive	Decomposition Temperature (°C) (°F)		Type of Reaction (5-s Test)
Nitrocellulose (NC)			
12.6% Nitrogen	170	338	Decomposes
13.45% Nitrogen	230	446	Decomposes
Nitroglycerine (NG)	222	432	Explodes
Pentaerythritol Tetranitrite (PETN)	235	455	Decomposes
Tetryl (TETR)	257	495	Ignites
Cyclonite (RDX)	260	500	Decomposes
Lead Styphnate	282	540	Explodes
Tetranitroresorcinol (TNR)	No data given		
Trinitrotoluene (TNT)	320-335 470	608-635 878	Explodes (confined) Decomposes (unconfined)

\*Data from "Engineering Design Handbook, Explosive Series, Properties of Explosives of Military Interest," AMCP 706-177, Headquarters, U.S. Army Material Command, January 1977



TABLE 4  
400 AREA SUMP AND VENT CONTAMINATION\*

Building	Explosive Residue	Heavy Metal Residue	Radiological Contamination	Vent Heavy Metal
402		X	X	
403	X		X	
404			X	
405		X	X	X
410				X
412			X	X
413		X	X	X
414		X	X	X
415	X		X	X
416				X
417	X	X		
418	X	X	X	
419	X	X	X	
421			X	X
422		X	X	X
423			X	
424			X	
425			X	X
426			X	
427	X		X	
430**			X	
431			X	
434			X	
435			X	
439			X	

\*Reported by Battelle

\*\*Sewer from Building 430 is contaminated by radiological material.

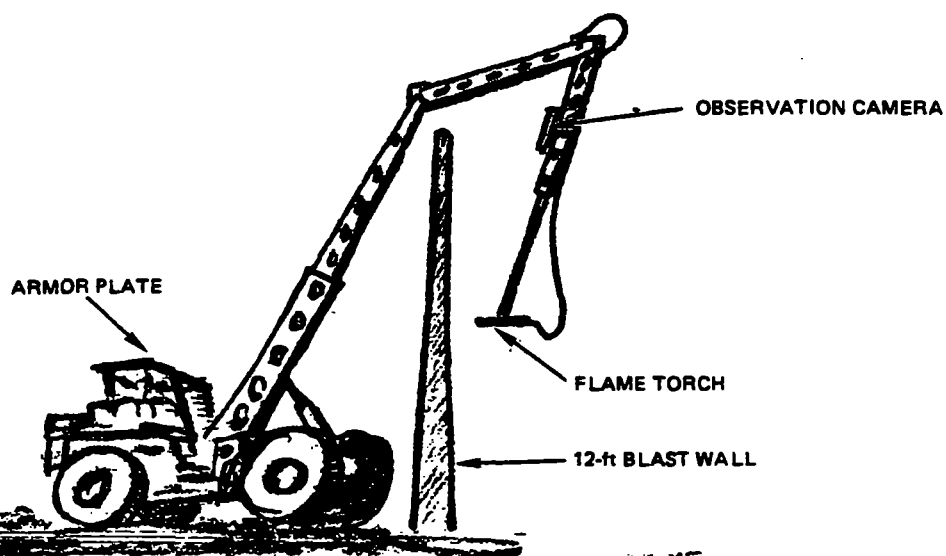


Figure 4. Remote Flame Machine

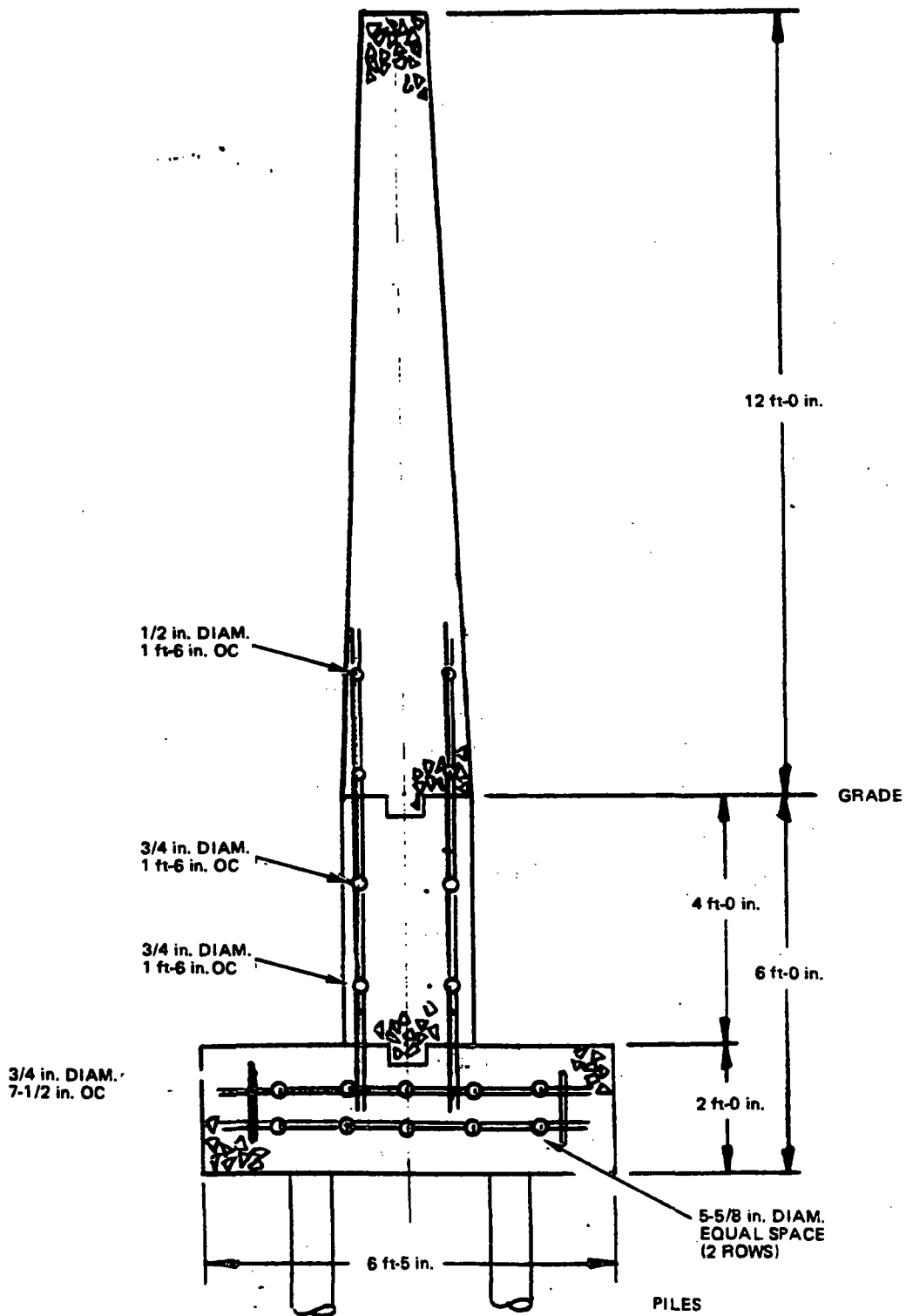


Figure 5. Typical Blast Wall Cross Section



RESTRICTED ACCESS AREA ENTRY PERMIT

Nº

UNIT AUTHORIZING OR PERFORMING WORK	REQUESTED BY _____ DEPT. _____	BEFORE STARTING WORK, SAFETY, SUPERVISION OF GROUP AUTHORIZING OR PERFORMING WORK, AND SUPERVISION OF WORK AREA MUST BE NOTIFIED				
	JOB LOCATION _____					
	JOB DESCRIPTION _____					
	REFERENCE PROCEDURE, WORK REQUEST, JOB ORDER, ETC. _____					
	DATE OF REQUEST _____ WORK TO BEGIN _____ WORK TO END _____					
SAFETY AND ORIGINATOR	NAME AND IDENTIFICATION OF PERSONS COVERED BY THIS FORM					
	NAME	NON-ROCKWELL AFFILIATION	INITIAL	NAME	NON-ROCKWELL AFFILIATION	INITIAL
SAFETY	<b>PROTECTIVE EQUIPMENT REQUIRED</b>					
	<b>HANDS</b>					
	<input type="checkbox"/> CANVAS GLOVES	<input type="checkbox"/> ACID APRON	<input type="checkbox"/> TOE GUARDS	<input type="checkbox"/> FULL-FACE RESPIRATOR		
	<input type="checkbox"/> NITROGEN GLOVES	<input type="checkbox"/> GONAD SHIELD	<input type="checkbox"/> CANVAS COVERS	<input type="checkbox"/> HALF-FACE RESPIRATOR		
	<input type="checkbox"/> PVC GLOVES	<input type="checkbox"/> HARD HAT	<input type="checkbox"/> PLASTIC COVERS	<input type="checkbox"/> ULTRA FILTER		
	<input type="checkbox"/> SURGEON'S GLOVES	<input type="checkbox"/> PHENOLIC	<input type="checkbox"/> BOOTS	<input type="checkbox"/> COMBINATION CANISTER		
	<input type="checkbox"/> LEATHER GLOVES	<input type="checkbox"/> FACE SHIELD	<input type="checkbox"/> LEGGINGS	<input type="checkbox"/> ORGANIC VAPOR		
	<input type="checkbox"/> GAUNTLET TYPE	<input type="checkbox"/> EYE PROTECTION	<input type="checkbox"/> JONES GOGGLES	<input type="checkbox"/> ACID GAS		
	<input type="checkbox"/> SAFETY GLASSES					
	<input type="checkbox"/> BODY					
<input type="checkbox"/> COVERALLS	<input type="checkbox"/> EAR PROTECTION	<b>RESPIRATORY PROTECTION</b>				
<input type="checkbox"/> LAB COAT	<input type="checkbox"/> EAR PLUGS	<input type="checkbox"/> SCBA				
<input type="checkbox"/> SURGEON'S CAP	<input type="checkbox"/> EAR MUFFS	<input type="checkbox"/> AIRLINE RESPIRATOR				
<input type="checkbox"/> HOOD	<input type="checkbox"/> SAFETY HARNESS					
<input type="checkbox"/> SAFETY BELT						
SAFETY	<b>PERSONNEL MONITORING EQUIPMENT -</b>		<b>SPECIAL REQUIREMENTS -</b>		<b>RADIATION MEASUREMENTS</b>	
	<input type="checkbox"/> BETA-GAMMA FILM BADGE	<input type="checkbox"/> PERSONAL SURVEY	<input type="checkbox"/> TOXIC ATMOSPHERE TEST		BETA _____ mrem/hr	
	<input type="checkbox"/> NEUTRON FILM BADGE	<input type="checkbox"/> TOOL SURVEY	<input type="checkbox"/> % LEL		GAMMA _____ mrem/hr	
	<input type="checkbox"/> BETA-GAMMA DOSIMETER	<input type="checkbox"/> WELDING PERMIT	<input type="checkbox"/> % TLV		NEUTRON _____ mrem/hr	
	<input type="checkbox"/> NEUTRON DOSIMETER	<input type="checkbox"/> CONFINED SPACE PROCEDURES	<input type="checkbox"/> STANDBY REQUIRED		TOTAL _____ mrem/hr	
	<input type="checkbox"/> EXTREMITY MONITORING	<input type="checkbox"/> OXY DEFICIENCY TEST	<input type="checkbox"/> FIRE PROTECTION		<b>SURFACE CONTAMINATION</b>	
	<input type="checkbox"/> PERSONAL AIR SAMPLER	<input type="checkbox"/> COMBUSTIBLE ATMOSPHERE			_____ dpm/100 cm <sup>2</sup>	
	<input type="checkbox"/> SURVEY METER	<input type="checkbox"/> % LEL			<b>AIRBORNE RADIOACTIVITY CONCENTRATION</b>	
					_____ µCi/mL	
	<b>SPECIAL INSTRUCTIONS</b> _____					
APPROVALS	<b>APPROVALS FOR PERFORMING WORK</b>					
	SUPERVISOR OF AREA WHERE WORK WILL BE PERFORMED _____					DATE _____
	SUPERVISOR OF GROUP AUTHORIZING OR PERFORMING WORK _____					DATE _____
	SAFETY _____					DATE _____ PERMIT EXPIRES _____

ALL PERSONS COVERED BY THIS PERMIT MUST INITIAL AFTER SAFETY INSTRUCTIONS HAVE BEEN GIVEN

AREA SUPERVISOR

APPENDIX A: RESTRICTED ACCESS AREA ENTRY PERMIT